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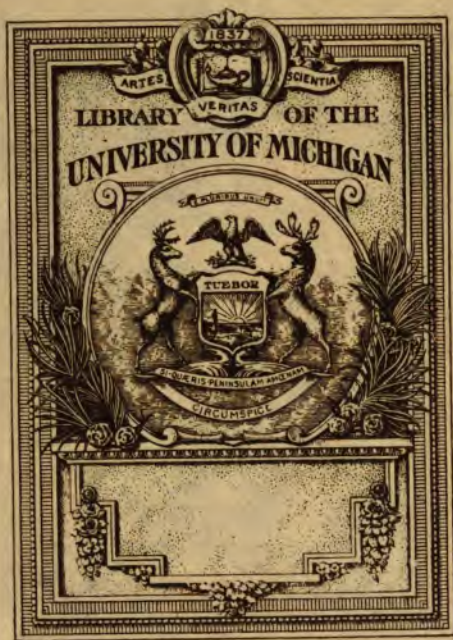
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**GENERAL INSTRUCTIONS AND METHODS
OF
ANALYSIS AND CHEMICAL CONTROL**

**FOR USE IN THE FACTORIES OF THE
CUBAN-AMERICAN SUGAR
COMPANY**

**CHAPARRA, DELICIAS, TINGUARO,
CONSTANCIA, UNIDAD, MERCEDITA,
REFINERY AT CARDENAS
REFINERY AT GRAMERCY, LA.**

**ARRANGED BY
GUILFORD L. SPENCER**

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GENERAL INSTRUCTIONS AND METHODS OF ANALYSIS AND CHEMICAL CONTROL

FOR USE IN THE FACTORIES OF
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INTRODUCTION

1. Introduction.—In order that the results obtained in several factories may be comparable, it is necessary that the methods of sampling and analysis in all be conducted by uniform methods. Different methods, notably in the analysis of low products, frequently give widely different results with the same material, rendering comparisons unsatisfactory. Authority shall be obtained from the Company before using modification of the methods specified in this pamphlet.

It is the purpose here to supply methods that are applicable in all the Company's factories, in obtaining the data required for the report sheets and permanent records. Methods for the routine control work are not given, as it is only essential that these supply figures for use in a single factory, and not for comparisons in a group of factories. Minor details of processes are omitted when strict uniformity is not essential.

The description of methods is preceded by definitions of terms as used by this Company and instructions to Superintendents relative to records and reports.

DEFINITIONS AND INSTRUCTIONS

2. Duties of the Chemist.—The chemist, under the general direction of the superintendent, is responsible for the chemical control. He shall personally assure himself of the integrity of all samples. He shall maintain system and good order in the laboratory. He shall promptly report defects in the manufacture, that may be detected through the control, to the superintendent. He shall keep full records of all reports and laboratory work. Verbal reports should later be reduced to writing and a carbon copy be retained among the chemist's records. All reports and communications in writing must bear the factory name and the date.

3. Mill Extraction.—This expression indicates the quantity of juice or sucrose extracted from the cane by the mills. It shall be expressed in percentage terms of the weight of the cane and the juice extracted, also in terms of the percentage of the sucrose in the cane that is obtained in the mill juice.

4. Saturation Water. **Saturation.**—The water sprayed upon the bagasse before regrinding is termed the "saturation," and shall be expressed in percentage terms of,

the weight of the cane. The words "maceration," "imbibition" and "saturation" are now usually used synonymously.

5. Bagasse.—The woody residue of the cane left after expressing the juice.

6. Dilution.—The dilution water is that part of the saturation water which is expressed with the juice, and mixing with it reduces its density below that of normal juice. The percentage of dilution is calculated from the densities of the normal and diluted juices and is reduced to terms of the weight of the cane. It is not always possible to obtain dilution numbers that represent only that water which is derived from the saturation. The engineers frequently cool the mill journals with water, a part of which may find its way into the juice pans.

7. Sucrose Retained. Retention.—This expression is designed to indicate the efficiency of the manufacturing processes; it shows the percentage of the sucrose in the raw juice, expressed by the mills, that is obtained in the commercial sugar.

In making low-grade sugar a higher retention number should be obtained than in the manufacture of white or other high-test sugars.

8. Boiling House Efficiency Number.—This is the percentage relation between the actual retention number and a theoretical number calculated by the Winter-Carp-Geerligs' formula $(1.4-40/P)100$, in which P is the coefficient of purity of the raw juice.

9. Sucrose, Polarization, Sugar, Glucose.—Sucrose is the pure carbohydrate of the formula $C_{12}H_{22}O_{11}$. Pure refined sugar is composed entirely of this carbohydrate. In commercial sugar work it is customary to use the word "sucrose" to indicate the apparent or approximate sucrose as ascertained by a direct polarization, rather than the true sucrose. The true sucrose content of a material is estimated by the Clerget method. The percentage is called the "true sucrose" or "Clerget number." "Polarization" is used to indicate the degrees on the cane sugar scale of the polariscope. The purer the material the nearer the polarization approaches the true sucrose. Many chemists use the word "sucrose" only for the carbohydrate and designate the apparent sucrose by "polarization." The customary commercial usage is followed in this pamphlet, using "sucrose by Clerget" or "true sucrose" to indicate the pure carbohydrate and "suerose" to indicate the carbohydrate as determined by direct polarization. The word "sugar" is usually used to designate the commercial product. "Glucose" indicates the invert and other reducing sugars that may be under consideration. All these are grouped under this word.

10. Available Sugar.—The amount of sugar that it is estimated may be obtained from the cane as indicated by the analysis is the available sugar. In the Company's factories this number is based upon the retention number or efficiency number that experience indicates may be realized in the particular factory. It is, therefore, the yield that may be expected.

11. Raw Juice, Normal.—The true normal juice is the juice as it actually exists in the cane. The juice cannot be separated from the cane in its normal state. By long-established usage, the juice obtained by milling, without the use of saturation water, is considered to be normal juice, and further, when saturation is practiced the undiluted juice from the first crushing is assumed to be of very nearly the density of the true normal juice and the mixed juice of its coefficient of purity. It is customary to apply a factor, deduced from experimental data, to reduce the density of the juice from the first crushing to a basis of the juice that would be obtained from all the mills without saturation. The density estimated in this way and the purity coefficient of the diluted or mixed juice are used in calculating the analysis of the so-called normal or undiluted

juice. The expression "normal juice" is used in this sense in the Company's records, and this use is sanctioned by long and very general usage in the cane-sugar industry.

It is evident that this expression indicates juice richer and purer than the average undiluted juice in the cane, and this is the more marked the more inefficient the mill work. There is no real need of the expression "normal juice" or of data based upon it.

12. Raw Juice, Diluted.—This expression indicates the mixed juices from all the mills, when saturation water is used. The diluted juice is the basis of the boiling-house control

13. Syrup.—The syrup is the purified juice after concentration; it is termed "meladura" in the Spanish-American factories.

14. First Massecuite.—The first massecuite is that produced largely or wholly from syrup or meladura.

15. Second Massecuite.—This massecuite is usually composed of a small quantity of syrup used as a nucleus or footing for the strike and molasses from a first massecuite. This may also be termed "mixed massecuite."

16. Third Massecuite.—This is a massecuite boiled, as described above, for a mixed strike, except using molasses from the latter, instead of from first massecuite. When the expression "mixed massecuite" is used, this massecuite may be termed "second" instead of "third."

17. Seed.—This is a magma prepared by mingling low-grade sugar with syrup or molasses for use as a footing or nucleus in starting a pan of sugar.

18. Molasses.—The molasses products are numbered to correspond with the massecuite from which produced. In a factory operating by the two-massecuite method, the second is the final or commercial molasses, and by the three-massecuite method this product is the third molasses. The molasses product of lowest purity is to be reported as final molasses.

19. Circulation Water.—The water used in condensing the vapors from the vacuum evaporating apparatus.

20. Residual Juice in the Bagasse.—The juice left in the bagasse at the completion of the milling process is termed "residual juice." The purity of this juice is used in calculating the percentage of fiber or marc in the bagasse and cane.

For the purposes of the Company's reports, the juice flowing from the bagasse roll of the last mill of the series is to be taken as the residual juice.

21. Fiber (Marc) in the Cane.—The water-insoluble matter of the cane.

22. Sucrose Per Cent Fiber in the Bagasse.—This is the percentage relation between two important constituents of the bagasse and has a bearing on the efficiency of the milling process.

23. Separate Days.—Each day of the season, beginning with the first grinding day and ending with the last, whether cane is ground or not, is to be entered as a "separate day."

24. Grinding Days.—Each day in which cane is ground is to be entered as a grinding day.

25. Lost Time.—Time is not considered lost except when the mills are forced to stop grinding through lack of cane, accident, etc. Only the time lost between the entry of the first cane of the season and the grinding of the last cane is to be considered. Time occupied in preliminary trials of the mills and in liquidating the house at the close of the season is not to be considered in the time report.

For convenience in checking the figures, the time elapsing between the beginning of the first grinding day and the actual hour of starting grinding need not be considered until the last day of the season, when it should be credited in the total lost time. It shall be entered upon the first report "to be credited at the end of season."

A specific cause must be given in accounting for lost time. For example, it is not sufficient to enter "6 hrs., lack of cane," the entry should show the cause of lack of cane, as "6 hrs., lack of cane due to rains," or "due to derailment" or other cause. "General cleaning" must not include stops for other causes, but solely the actual time required for cleaning. The chemist charged with the record work must investigate the causes of stoppages if not clearly shown in the time reports and properly record them.

26. Decimal Entries.—While it is conceded that many of the analyses, etc., cannot be correct to two decimal places and sometimes not even to one, entries in the Run Reports should be made to two places and also in the Daily Reports, except control tests of molasses, which should be to the nearest tenth.

27. Distribution of Reports.—Reports are designed primarily for the use of The Cuban-American Sugar Company. A list of officials who should receive these will be upplied to each laboratory. A copy of the distribution list of each factory, together with additions and amendments, shall be filed with the Chief Chemist.

STANDARDIZATION OF SPECIAL APPARATUS

28. Hydrometers.—As the Brix hydrometer indicates percentages of sucrose by weight in solutions of the pure carbohydrate, it may readily be checked by the polariscope. Three sugar solutions should be prepared with distilled water and pure refined sugar, for use in checking the hydrometer at three points. The percentages of sucrose should be determined in these solutions by the polariscope, using standardized instruments. The hydrometer must be thoroughly cleansed before standardizing. All traces of oil from the hands or other source must be removed and only the upper end of the stem should be touched by the hands after cleaning. The hydrometer cylinder should be filled to the brim with the solution so that this will overflow and carry off bubbles or dust when the hydrometer is inserted. The temperature of the instrument should be near that of the sugar solution. The solution should be gently and thoroughly stirred before inserting the instrument and both the liquid and the hydrometer should be free from air bubbles before reading the scale.

The hydrometer, when floated in these solutions at a temperature of 17.5° C., should, if correct, indicate percentages agreeing with those ascertained by the polariscopic method. If the observations are made at other than 17.5° C., the readings must be corrected by the tables in current use. The errors of the hydrometer at the top, middle and bottom parts of its scale should be noted. This standardized hydrometer may be used in checking other hydrometers. The hydrometers should be numbered or bear other marks for identification. Diamond ink is convenient for marking these instruments and other glassware.

29. Volumetric Glassware.—The glassware should be thoroughly cleansed first with an alkali solution, then with nitric acid and finally with a warm solution of chromic acid in sulphuric acid, followed by rinsing with distilled water. The flasks selected should have necks of small internal diameter, preferably about 12 mm. They should be thoroughly dry and should remain inside the balance case a few minutes after wiping them, before weighing. The flask should be carefully examined for traces of moisture and

this be eliminated before weighing. Recently boiled distilled water, cooled to the room temperature, should be used in calibrating glassware in the factory laboratory.

A small error is necessarily permissible in graduated ware. The error should be below 0.05 cc. for a 100-cc. flask or 0.03 for a 100-cc. pipette, and less or smaller apparatus.

30. Standardizing a Flask.—Counterpoise the dry flask on the balance pan. Remove and fill the flask to the mark with the distilled water, using a pipette in filling, and avoid wetting the neck of the flask or carrying bubbles of air into it. This manipulation requires great care. For small flasks, the analytical balance and accurate weights must be used. The weighing should be by substitution. The following table shows the number of grams of water that 100-cc. Mohr's flask should hold at various temperatures, as weighed with brass weights in the air:

APPARENT WEIGHT OF WATER CONTAINED BY A 100 CC. MOHR'S FLASK
AT VARIOUS TEMPERATURES

C°	Grams	C°	Grams
15	100.033	24	99.879
16	100.020	25	99.857
17	100.007	26	99.834
17.5	100.000 = 100.234 true cc. at 17.5°	27	99.809
18	99.991	28	99.784
19	99.975	29	99.758
20	99.958	30	99.731
21	99.940	31	99.704
22	99.921	32	99.674
23	99.900	33	99.644

In the above table allowance is made for the expansion of the glass. If, for example, the flask holds 99.9 grams of water at 23° C., its capacity is 100 cc. (Mohr), and it is correct for use with a normal weight of 26.048 grams. A standardized pipette is convenient for use in checking flasks, enabling one quickly to reject those that are outside the limits of tolerance.

Polariscopes are now graduated at a standard temperature of 20° C., and according to the specifications of the International Committee to be used with a normal weight of 26 grams and a flask graduated to hold 100 true or metrical cubic centimeters. Owing to the increasing difficulty in procuring ware graduated to Mohr's units, the Company will ultimately conform with the specifications of the International Committee. The tables on page 8 are, therefore, inserted.

The tables of corrections give for each nominal capacity and observed temperature the amounts to be added to the apparent weight (in air against brass weights), of the water contained by the flask to give the capacity in true cubic centimeters at 20° C. Example: Apparent weight of water at 23.5° C. held by a 100-cc. flask is 99.63; adding the correction 0.35, corresponding to 23.5° C., we have $99.63 + 0.35 = 99.98$, the actual capacity in true cc. at 20° C.

31. Standardizing Burettes and Pipettes.—Burettes and pipettes are standardized by weighing the quantity of water they deliver. A flask is used in making the weighings. The tables of weights of water that have been given are also used in checking

TABLES OF CORRECTIONS FOR DETERMINING THE TRUE CAPACITIES OF FLASKS FROM THE WEIGHT OF WATER IN AIR

(From Bul. No. 19, U. S. Bureau of Standards.)

(Following data are assumed as approximating ordinary conditions: Observed barometric pressure = 76 cm.; relative humidity = 50 per cent; coefficient of expansion of glass = 0.000025 per degree C.).

INDICATED CAPACITY 50 CC.

Temp. Deg. C.	Tenths of degrees									
	0	1	2	3	4	5	6	7	8	9
15	0.104	0.104	0.105	0.106	0.106	0.107	0.107	0.108	0.109	0.109
16	.110	.111	.111	.112	.113	.113	.114	.115	.116	.116
17	.117	.118	.118	.119	.120	.121	.121	.122	.123	.124
18	.124	.125	.126	.127	.128	.128	.129	.130	.131	.132
19	.132	.133	.134	.135	.136	.137	.137	.138	.139	.140
20	.141	.142	.143	.144	.144	.145	.146	.147	.148	.149
21	.150	.151	.152	.153	.154	.155	.156	.157	.158	.159
22	.160	.161	.162	.163	.164	.165	.166	.167	.168	.169
23	.170	.171	.172	.173	.174	.175	.176	.177	.178	.179
24	.180	.182	.183	.184	.185	.186	.187	.188	.189	.190
25	.192	.193	.194	.195	.196	.197	.199	.200	.201	.202
26	.203	.204	.206	.207	.208	.209	.210	.212	.213	.214
27	.215	.216	.218	.219	.220	.222	.223	.224	.225	.226
28	.228	.229	.230	.232	.233	.234	.236	.237	.238	.240
29	.241	.242	.244							

INDICATED CAPACITY 100 CC.

Temp. Deg. C.	Tenths of Degrees.									
	0	1	2	3	4	5	6	7	8	9
15	0.207	0.208	0.210	0.211	0.212	0.213	0.215	0.216	0.217	0.219
16	.220	.221	.223	.224	.225	.227	.228	.230	.231	.232
17	.234	.235	.237	.238	.240	.241	.243	.244	.246	.247
18	.249	.250	.252	.253	.255	.257	.258	.260	.261	.263
19	.265	.266	.268	.270	.272	.273	.275	.277	.278	.280
20	.282	.284	.285	.287	.289	.191	.293	.294	.296	.298
21	.300	.302	.304	.306	.308	.310	.311	.314	.315	.317
22	.319	.321	.323	.325	.327	.329	.331	.333	.336	.338
23	.340	.342	.344	.346	.348	.350	.352	.354	.357	.359
24	.361	.363	.365	.368	.370	.372	.374	.376	.379	.381
25	.383	.386	.388	.390	.392	.395	.397	.399	.402	.404
26	.406	.409	.411	.414	.416	.418	.421	.423	.426	.428
27	.431	.433	.436	.438	.440	.443	.446	.448	.451	.453
28	.456	.458	.461	.463	.466	.469	.471	.474	.476	.479
29	.482	.484	.487							

Having properly arranged the polariscope and lamp, the zero point must be checked by a blank observation, preferably with a tube of water in the trough. The vernier must be moved with the adjusting key until the instrument indicates zero under the above conditions. After setting the vernier, a standard quartz plate, reading approximately 96° , should be substituted for the tube of water and an observation made. The deviation from the marked value of the plate should be noted and allowed for in all observations. The eyes of different observers may differ, hence each should make

check observations on the quartz plate and apply the proper corrections in his analytical work. The plate must not have been wiped or pressed within several hours of using it for checking.

The eye should be allowed time to adjust itself to the instrument and recover from fatigue between tests. An observation should not be made immediately after looking at a bright light. A plate of colored glass should be placed in front of the lamp to protect the eyes when lighting it.

The observation tubes should be compared with one another by polarizations using the same sugar solution, or they should be carefully calibrated. The cover glasses should be examined frequently, and if their surfaces are scratched or not parallel, or if they show optical activity, they must be rejected. Care must be observed that the caps do not press unduly on the cover glasses, since glass becomes double refracting under pressure.

Each laboratory should be provided with a standard quartz plate, certified normal weights, and thermometer. These should be used only in checking the apparatus and not in the analytical work. Polariscopes and weights should be occasionally checked against the standards in possession of the Company's Chief Chemist, who will supply these on application.

WEIGHTS AND MEASUREMENTS

33. Weights and Measurements.—The chemist must assure himself that all weights and measures are of one system, *e.g.*, all Spanish, metric or English; if not, they should be reduced to one system before proceeding with the control calculations. The accuracy of all scales and the calibration of all measuring tanks should be verified by the chemist. Spanish weights are usually used in Cuba, of which one hundred pounds Spanish = 46.0096 kilograms $\therefore$ 1 lb. Sp. = 1.0143 lb. avoirdupois. This equivalence is in accordance with the decree of a Spanish Captain General in Cuba.

34. Weight of the Cane.—If the extraction figures are inconsistent an error in the weight of the juice or cane is probable. If the error is not promptly detected, all figures affected shall be included in the daily reports with the indication, "Subject to correction," and the report shall be issued. When the error is located, an additive or subtractive correction shall be entered in the unit book, between the lines, in red ink. Each day's report should be considered as closing the business of the day. An error of one day must not be permitted to affect the figures of the next or a subsequent day. All laboratory entries and figuring should be carefully checked before raising the question of an error with the Cane Department.

35. Measurement and Weight of the Juice.—The Company's factories are all equipped with tank scales for weighing the juice. The following description is for use in the event of a scale being out of commission.

Where the juice cannot be weighed, its weight must be deduced from its volume and density. Where practicable, the volume of the measuring tank should be determined by running water into it from an accurately calibrated tank. This is not always practicable in large factories, hence the volume must usually be calculated from the average of several measurements.

An overflow pipe should be provided in the tank. This should terminate inside the tank in the side branch of a tee. The lower branch of the tee should be extended with a nipple a few inches downwards. The upper branch should also be extended that it may always be open above the foam on the juice. The overflow should be led back to

the mills. The object of the tee is to prevent foam from clogging the outlet to the overflow pipe. The liming tanks should be used in measuring the juice.

The juice entrains a large quantity of air and some fiber which increase its volume. This introduces an error for which a correction based upon experimental data must be made. The volume of air entrained varies with the temperature of the juice and other conditions. The weight of the juice may be deduced from the volume as described on page 34 under Mill Extraction.

36. Measurement of the Saturation Water.—Two tanks should be used in measuring the water, one to be filling while the other is emptying, or one measuring and one receiving tank may be used. The weight of the water is calculated from its volume. Meters are often unsatisfactory for this purpose. Time sheets should be used, noting the time of filling or discharging a tank, as a control on the measurements.

An error in the measurement of the water affects the sucrose extraction number and the calculated percentages of sucrose and fiber in the cane since it affects the percentage of bagasse.

If possible the water should be weighed. If measured by meter, this instrument should be standardized while measuring water of the temperature of that used in the saturation. The table on page 33 may be used in calculating the weight of the water from its volume.

Where, through accident or other cause, the volume of the water cannot be determined, it must be estimated from the dilution of the juice. This is necessary in order to estimate the weight of the bagasse. This method of estimation is purely arbitrary. The dilution varies with the quantity of saturation water and the efficiency of the milling. The leakage of water from mill journals must also be considered. The dilution number varies from below 0.7 with poor milling to above 0.9 of the saturation number with very good mill work. An inferential number calculated from the analytical data is usually very unreliable.

37. Filter Press-cake.—The cake should be weighed, but as this is not usually feasible, its weight may be estimated from that of several average cakes. The number of presses emptied should be recorded for use in calculating the total amount of press cake. Where the cake is dropped into cars or carts and weighed, care must be observed to avoid including water from drip and floor washings in the weight.

Where double filter-pressing is practised, and the cakes contain but a small fraction of a per cent of sugar, the errors of estimate of weight and sampling become of minor importance.

38. Masecutes and Molasses.—Masecutes should be measured immediately after they are discharged from the pans. This is necessary on account of the expansion this material undergoes in the crystallizers. These measurements are not required except preparatory to a "Run Report."

It is usually difficult accurately to measure final molasses. This is due to the air it entrains. Where the molasses is slightly reduced with water and steam to bring it to a uniform density, it may be measured before pumping if from the factory, or afterwards in a large storage tanks. Such dilution is forbidden in the present molasses contracts of the Company. No water should be added to the molasses without authority from the Company. Measurements of very heavy molasses in tank cars are often unsatisfactory even when the measurement is made several hours after filling the tanks.

For control purposes, especially, for the final report of the Crop, the actual weight

of the molasses should be reported, though, in order not to delay the reports, a small quantity may be estimated. The tank-cars should be tared each trip because the buyer sometimes does not completely empty them.

Where measurements are necessarily made of heavy molasses in large tanks or tank-cars, corrections determined by experiment are required for the entrained air. Heavy molasses will often entrain sufficient air to reduce its weight per gallon nearly or quite one pound. Where apparent measurement alone is depended upon, this error may cause the "undetermined" or apparent loss of the sucrose balance to almost or quite disappear.

SAMPLING AND AVERAGING

39. Preliminary Remarks.—Sampling should be so conducted that the quantity of material drawn from each tank, etc., bears the same relation to the total quantity. For example, having two equal tanks of syrup, one full, the other half full, half as much should be drawn from the one as from the other, that the subsamples, when mixed, may represent all the syrup. This principle should be applied in all sampling.

In averaging analytical data, each analysis shall be weighted by the quantity of material it represents, or in other words, the weight of each constituent shall be calculated and the sum of the weights be divided by the total weight of the material.

The chemist shall confer with the superintendent and arrange to avoid an unnecessary multiplication of samples and analyses. There is a tendency in factories to make several tests, averaging the results, where a single test on a composite sample will answer every purpose equally well and usually is more accurate. There should be a definite purpose in each sample and in each analysis.

40. Cane.—Small samples selected in the fields and at the mills are usually of little value in judging the general character of the cane. This is due to the wide variations found in a field and even in a single stool. When possible the sampling should be performed at the mill and the sample should include several cartloads or a railway car of cane. The method of analysis should be indirect, *i.e.*, the richness should be calculated from the analyses and weights of the juice and bagasse. A sample of juice should be drawn at the crusher or first mill for the density determination and the mixed juices should be analyzed to determine the purity, and from these two numbers the sucrose may be calculated. By exercising care, the test sample can be drawn after the juice from the cane to be sampled has washed the mills and pans.

41. Juice.—The ideal sample is one drawn continuously in proportion to the output. The Calumet automatic sampler draws a sample under approximately these conditions. This instrument resembles a vacuum-pan proof-stick and is operated by some part of the milling machinery, preferably a roll shaft. It is actually a very simple pump. It is convenient to have duplicate Calumet samplers, one for drawing the regular and the other for the special samples. Drawings of these samplers will be supplied to the laboratories on application to the Chief Chemist.

When using tank scales, the juice may be sampled by drawing a small cupful from each charge, to be composited to represent a run of definite duration, preferably a watch of six hours or a day. The sample shall not be poured through a funnel into the storage jar. The jar shall have a very wide mouth and the sub-sample shall be poured directly into it. The cover should be replaced on the jar after every addition of juice. In a single tandem house it is preferable to draw the sample from the pipe line with a pump-sampler.

Difficulty is sometimes experienced in sampling in a factory having two or more tandems of mills owing to the differences in the rate of grinding and of the efficiency of the milling plants. If the juice lines converge to a single pipe near the liming tanks, an automatic sampler may be used, but care must be observed to avoid an error when one set of mills is stopped. In this event the speed of the sampler should be reduced to correspond with the number of mills operating. It is safer to draw a measure of juice from each scale tank.

A sample of the juice must be drawn from the crusher canal and one of the mixed juices as described above. The sample from the crusher canal is more representative than one drawn from the juice canal of the crusher and first mill would be. There is no certainty that the sample from the mill canal is thoroughly mixed and further where water is used in cooling the mill journals some of it may enter the juice pan and canals.

The sample from the crusher or first mill is for the determination of the density and that of the mixed juice is the basis of the factory control.

The density samples should be treated with a 40 per cent solution of formaldehyde or mercuric chloride, one part of either to 5000 parts juice, for preservation. The mixed juice sample should be drawn in duplicate, one portion (A) to be preserved with about 12 grams Horne's dry subacetate of lead per litre and the other (B) with either formaldehyde or mercuric chloride. No formaldehyde should be used in the (A) sample.

It is a good practice when the mill work is uniform to draw a single sample of mixed juice at the mills or a small cupful from each scale tank of juice and composite this at hourly intervals in the laboratory, making two composite samples, preserving one with dry lead and the other with formaldehyde.

Where continuous samples cannot be obtained, small equal-measured subsamples must be drawn and composited. Only sterilized, wide-mouthed jars with stoppers or covers are to be used for storage purposes. Funnels must not be used to direct the sample into the jar. The small sampling cups should be thoroughly rinsed with the juice before drawing the subsamples. The sample must be mixed with the preservative after each addition of juice. Samplers and recipients must be frequently sterilized.

42. Bagasse.—Subsamples should include all the bagasse on one or more slats of the conductor. Composite samples, well protected from the air, may be preserved during a few hours in an atmosphere of formaldehyde, but the storage period must not exceed six hours. Clean galvanized-iron cans are to be used for storing the samples. These shall have a small compartment to hold cotton or a sponge saturated with formaldehyde. The slip-cover of the compartment should be perforated with a number of holes.

With the heavy milling now practiced in the Company's plants, the bagasse as it leaves the mills is usually sufficiently comminuted for the moisture test in the special drying ovens (*see Bagasse*). It should be remembered that the loss of moisture in the sample introduces a very sensible error in the estimation of the moisture content of the bagasse. Mix the bagasse thoroughly but do not attempt to comminute it. Subsamples should be collected every hour or more frequently and be composited for an analysis every six hours.

The sample for the sucrose test should be cut into small pieces in a chopper. The loss of moisture introduces an error that probably compensates in a measure for the slightly imperfect extraction of the sucrose in the analysis, provided the loss is not excessive.

43. Residual Juice of the Bagasse.—The juice flowing from the bagasse roll of the last mill probably corresponds so nearly with the true residual juice that it may be accepted as such. A sample shall be drawn, with a wire sampler or a long-handled cup, at the time of sampling the bagasse. It shall be subsampled and composited as has been directed for juice.

44. Filter Press-cake.—Subsamples should be cut from a definite number of cakes and from representative parts in each press by means of a special sampler. This is a brass receptacle, small enough to grasp in the hand and provided with a tubular cutter. The receptacle contains a sponge cup for formaldehyde. The cutter should be made of a slightly conical brass tube, the large end connected with the receptacle. The cutting edge should be very blunt, usually about one-eighth inch thick, to avoid injuring the filter cloths. The receptacle should have a cover fastened with a bayonette catch. A sponge cup, for cotton, saturated with formaldehyde, should be attached to the inner side of the cover. The small samples could probably be safely collected during a very few hours without recourse to a preservative, but formaldehyde should always be used.

45. Syrup and Molasses.—Syrup and thin molasses, to be reboiled, may be sampled in the storage tanks. Heavy commercial molasses should be sampled at frequent regular intervals at the pump-tank, forming a composite sample, representing the day's work. When shipping molasses, samples shall be drawn from each tank-car, to form a composite sample representing the day's shipment. Composite samples should be prepared from the daily samples to form fortnightly samples for analysis to supply the data required in the Run Reports.

46. Masseccutes.—Masseccutes are difficult to correctly sample owing to the variations in different parts of a "strike." Where this material can be sampled as it flows from the pan, small portions should be drawn from time to time to form a composite sample, but where discharge pipe lines are used this method is not feasible. In the latter case the sample should be obtained with the proof-stick or from the crystallizer or mixer, being careful to avoid including steamings and to allow time for thorough mixing.

When sampling from pans of different sizes, to form a composite sample representing a definite period for all, small conical tin measures should be used, one for each size of pan, representing the same proportional part of the material.

First masseccute samples may usually be composited for analysis once daily. The analyses of the second and third masseccutes are usually required promptly to control the molasses and its injection into the pans.

47. Sugars.—One hundred per cent of the packages must be sampled. As the truck passes onto the scale, or as the bag leaves the automatic scale, a spoonful of sugar should be withdrawn and thrown into a zinc-lined box, provided with a small funnel or hopper-like opening. Sugar already packed should be sampled with a trier.

A composite sample, in proportion to the output of sugar, should be prepared from the daily samples for analysis for the run reports, but samples should not be composited longer than a fortnight.

48. Remarks on Sampling.—The principles described apply in all sampling and averaging in the control of the factory. Where practicable, composite samples should be prepared to supply data for the permanent records and daily reports, so that but a single analysis need be made daily of each class of material for these purposes. The pan control and mill control, however, require frequent analyses of masseccutes, molasses and bagasse.

All sampling devices and containers must be kept scrupulously clean. The chemist

is responsible for the efficient working of sampling devices, their cleanliness and the cleanliness of the container. If samples are not representative of the materials from which they are drawn, they are worthless and the control becomes a failure.

METHODS OF ANALYSIS

49. Sugar Cane.—Sucrose.—Owing to its nature, the cane cannot be accurately sampled on a small scale without undue expense for appliances and labor. For this reason and not on account of analytical difficulties, its analysis should be ascertained indirectly as follows: The per cent sucrose in the cane = (weight of sucrose in the raw juice ÷ weight of sucrose in the bagasse) ÷ weight of the cane × 100.

Fiber.—The trash, as well as the fiber, influences the milling and fuel supply, hence it is preferable to deduce the per cent fiber from the bagasse tests rather than by direct analysis of the cane. Care should be observed that only water derived from the saturation is included in the dilute juice. Direct analysis for fiber is also open to the objections already stated in the paragraph on cane sampling. Further, the calculated percentage of fiber, in a well-controlled factory, probably approximates the true per-centage more closely than the number by direct test.

The per cent fiber, or marc, in the cane = fiber per cent bagasse × bagasse per cent cane ÷ 100. (See Bagasse Analysis.)

50. Juice.—Brix.—Strain the sample, through fine centrifugal lining, into a tall cylinder, letting the latter overflow. After an interval of about ten minutes insert the hydrometer, at the same time blowing the foam and impurities from the surface of the liquid. After the instrument reaches the temperature of the juice, note the graduation on its stem on a level with the surface of the juice in the cylinder, and enter this number as the observed degree Brix. The temperature of the juice should also be noted and a correction be made by Gerlach's table, page 39, if it varies from 17.5° C.

Sucrose.—Thoroughly mix the sample of juice that has been preserved with dry sub-acetate of lead and filter a portion of it. Polarize the filtrate and calculate the percentage of sucrose, using Schmitz's table. If the Schmitz table providing for 10 per cent dilution only is available, divide the polariscope reading by 1.1. A copy of Schmitz's table, for use without dilution, will be supplied to each of the Company's laboratories, thus obviating the division by 1.1.

The estimation of the sucrose by the double polarization or Clerget method is conducted in general as is described on page 18 for final molasses. If the modified Herzfeld method is used 75 cc. of de-leaded clarified juice may be inverted in a special inversion flask. If working by the Steuerwald method (30 cc. acid), invert 70 cc. of juice or less if the polarization is above 18°. Clerget tests should be made at frequent intervals of composite samples of the mixed juices preserved with mercuric chloride, using normal acetate of lead for clarifying, or the dry lead may be used and afterward precipitated with sodium oxalate. This procedure is advisable on account of the precipitation of dextrose and levulose in the presence of certain salts by the basic-lead salt. These analyses are for use in estimating the Clerget test of the average juice for the Run. If these tests indicate a certain increase or decrease in the sucrose as compared with the direct polarization, the same percentage of change is to be applied to the average polarization, of the raw juice to estimate its Clerget number.

Glucose.—The glucose is preferably determined in a sample that has been clarified with normal acetate of lead. This is feasible with fresh samples. On account of the

necessity of a preservative in compositing samples, the basic and not the normal acetate must usually be used. After filtering off the lead precipitate in the clarification, delead the filtrate by the addition of sodium oxalate in very slight excess, allowing time for the reaction and repeatedly testing filtrates to avoid more than a very slight excess of the oxalate. Wedderburn advises the use of a phosphoric acid solution, *e.g.*, a 10 per cent solution of the acid in deleading. This is a permissible and probably preferable deleading agent. Preliminary tests in the Control Laboratory indicate phosphoric acid to be the best deleading agent.

Proceed with the reduction, etc., as is described on page 19 for molasses.

Solids by Drying.—See Methods of Drying (58).

Ash.—See Ash Determinations (58).

51. Bagasse.—*Sucrose.*—Digest 100 grams of bagasse, reduced to small fragments, in a suitable vessel, such as a double boiler as is used in the kitchen with 900 cc. of water and 5 cc. of a 5 per cent solution of sodium carbonate, during one hour at near boiling temperature in a water or steam bath. Stir well and frequently. After digestion cool and weigh the vessel and contents. Drain off a portion of the liquid, clarify it with dry subacetate of lead, and polarize, using a 400-mm. observation tube.

A tall Kodak solution cup or tank is very well adapted for this digestion. The 2½ inch size is suitable. A small opening should be made in the cover, and a piece of small bore rubber tubing inserted. A glass rod should close the rubber tube during the digestion and should be removed while cooling the cup and contents. A steam or water bath should be used in heating. If a bath is not available, a reflux condenser should be used to return the condensate to the cup. The temperature should not quite reach boiling. A tamp or mixer should be placed in the cup, if used with a condenser. This should have a long handle extending through the condenser tube and be frequently used in mixing the contents of the cup. If the tamp is not used, the bagasse should be prevented from floating by placing a perforated plate upon it. In using the Kodak cup the bagasse and solution should be thoroughly mixed by shaking.

Example and Calculations:

Weight of bagasse used.....	100	grams
Fiber in the bagasse.....	48	per cent
“ “ “ sample.....	48	grams
Weight of digestion beaker + bagasse + water.....	1248.5	grams
“ “ “ “	243.5	“
“ “ “ bagasse + water.....	1005	“
“ “ “ fiber in bagasse.....	48	“
“ “ “ extract.....	957	“

Clarify a portion of the extract with Horne's dry subacetate of lead and polarize, using a 400-mm. observation tube. Polariscopes reading of the extract, using a 400-mm. tube=2.6. The extract is so dilute that its density need not be considered in the calculations. A correction must be made for the use of a 400-mm. tube by dividing the polariscopes reading by 2. By Schmitz's table, the reading 2.6 corresponds to 0.67 per cent sucrose (the per cent sucrose in the extract is half this number), and the grams of extract, 957×0.34 per cent = 3.25 per cent sucrose in the bagasse.

At least four sucrose tests, representing the four watches, shall be made daily, of samples of bagasse from each train of mills.

Moisture.—The moisture tests should be made in the Company's special oven. This is a device for drawing heated air through the sample contained in a tared tube, one end of which is closed with perforated centrifugal sheet and the other with a removable cover of similar material. The draft is produced by a steam ejector. The time required for complete drying will not exceed 90 minutes at a temperature of 110° C. The temperature may advantageously and safely reach or exceed 135° C., and with increased rate of drying. The volume of hot air drawn through the sample should be very large.

Small shallow trays made of 80-mesh brass-wire gauze may be used in the moisture determinations in the absence of the special oven. These should hold at least 100 grams of bagasse and should be narrow enough for weighing on the sugar balance. The bagasse may be dried at 110° C., but preferably 135° C., until there is no further loss of weight, or until there is a gain after a short interval, the lowest weight being accepted. The dry bagasse absorbs moisture with extreme rapidity, therefore, the weighings must be quickly made. The approximate weight of the cylinder or tray and dry bagasse should be placed in the balance pan before removing the dry bagasse from the desiccator.

The bagasse should be prepared for the moisture test by chopping only when it is very coarse. It is not usually advisable to attempt to reduce it for the moisture test, especially if it has been well broken up by good milling, on account of the long exposure to the air that would be necessary. At least four samples of bagasse from each train of mills, representing the four watches, shall be dried daily.

Residual Juice.—The juice flowing from the bagasse roll of the last mill may be accepted as of the purity of the residual juice. A sample of this juice shall be drawn at the time of sampling the bagasse, and the coefficient of purity of a 24-hour composite sample determined.

Fiber (Marc).—The fiber shall be estimated by the indirect method. The calculation by this method is made as follows: Percentage sucrose in bagasse ÷ coefficient of purity residual juice × 100 = per cent juice solids in bagasse; 100 - (per cent moisture in bagasse + per cent juice solids) = per cent fiber.

52. Filter Press-cake.—**Sucrose.**—Reduce the sample to a fine state of division. This is readily accomplished, provided the presswork is satisfactory, by cutting the sample in a large mortar with scissors. The scissors should be held with a handle in each hand.

Rub 50 grams of the press-cake in a mortar to a cream with water and wash it into a 200-cc. flask, Stiff's or Kohlrausch's form. Add sufficient subacetate of lead to clarify the solution and dilute it to the mark with water. Shake the flask and contents very thoroughly and filter. Polarize the filtrate, using a 200-mm. tube. The polariscope reading is the percentage of sucrose in the sample.

The following is a convenient method of compositing filter-cake samples: Rub 25 grams of the six-hour sample to a cream in a mortar with water and 6 cc. subacetate of lead solution and set this aside until the next six-hour sample is ready. Add 25 grams of the second sample and 6 cc. of the subacetate to the previous sample and again rub to a cream. Continue in this way until the fourth sample has been added and then wash the contents of the mortar into a 200-cc. flask and proceed with the analysis as already described.

Moisture.—Dry 5 grams as directed under Methods of Drying (58). The moisture

is only required in special investigations, e.g., estimation of water in diluting cachaza, and not in routine work.

53. Massecuites.—Brix.—Dissolve a weighed portion of the material in an equal weight of water and determine the corrected degree Brix of the solution; multiply this number by 2. It is important that the sample be thoroughly mixed and that all crystals be dissolved.

The Brix, as determined in this way, is a commercial number and is for use in comparisons. There are certain conditions which prevent the number from being a true one. The method should not be modified, except in special research work.

Sucrose.—The direct polarization only is usually required. A portion of the solution for the Brix test should be used. Solutions of first massecuites may usually be clarified with normal lead acetate solution. If the clarified solutions of first or other massecuite using lead acetate, are too dark, use subacetate of lead and proceed with the direct polarization as is described farther on for final molasses. The Clerget test, if required, is made as in the final molasses.

In the control of the pan boiling, a convenient and sufficiently accurate method of analysis is as follows: Reduce the material to approximately 20° Brix with hot water; cool the solution by passing it through a coil of three-eighth inch tin tubing immersed in a stream of cold water; determine the Brix of the solution and measure the quantity corresponding to it with a sucrose pipette into a flask and proceed as usual with the analysis, and calculate the purity. If an approximate Brix of the original material is required, determine the sucrose in it and from the two sucrose numbers and the Brix of the dilute solution calculate the Brix of the original material. It must be considered that analyses of dilute solutions give lower purities and higher Brix than by the usual method. This is immaterial in pan control provided one method is used for all these tests that the data may be comparable. In a factory that has been using the usual method a few comparative tests should be made that the superintendent may note the differences by the two methods and take them into account.

Glucose.—Ash and Moisture.—See 55 for Glucose and 58 for Ash and Moisture.

54. Molasses.—Brix and Polarization.—Proceed as has been specified for massecuites.

55. Final Molasses.—Degree Brix.—Proceed as has been described for massecuites.

Polarization and Sucrose by Clerget Method.—All polarizations of molasses shall be made in acid solution. Samples must be well mixed as has been described under "Degree Brix" and "Glucose Determinations." Use the solution prepared for determining the "Degree Brix" or an equivalent quantity of the molasses in preparing the solutions for the polariscope.

Direct Polarization.—Weigh 5 normal weights of the molasses solution prepared for the Brix test—equal weights of molasses and water—or 2.5 normal weights of the molasses itself, and wash this into a 500-cc. flask. Add 60 cc. of 54.3° Brix subacetate of lead solution, dilute to the mark and mix thoroughly by shaking and then filter. It may occur that 60 cc. of the lead solution is insufficient for proper clarification. If so, more may be used, but this additional quantity must be ascertained by careful trial. The chemist shall ascertain the quantity needed and require his assistants to proceed accordingly. There is usually very little variation during a crop in the quantity of lead necessary. Errors in molasses tests are sometimes traceable to lack of solution of the fine crystals that may be in sample or to incomplete mixing of the solution and precipitant.

Measure 50 cc. of the filtrate in a 50/55 cc. flask and add nearly 5 cc. of 5 per cent acetic acid or sufficient to acidulate the molasses solution and dilute with water to 55 cc. Polarize the solution in a 200 mm. water-jacketed observation tube (Landolt tube), with water circulation. Note the temperature of the observation. To correct for the use of a half-normal solution and the acetic dilution, multiply the polariscope reading by 2.2, to ascertain the direct polarization, D of the formula.

Invert Polarization. Steuerwald's Method.—Either of the following methods is optional: To 200 cc. of the filtrate for the direct polarization add 4 grams of sodium oxalate, mix and allow to stand 30 minutes. Add a small quantity of dry kieselguhr (filtercel), mix by shaking, and filter. Measure 50 cc. of the filtrate with an accurate pipette¹ or burette, into a 100 cc. flask.

Add 30 cc. of hydrochloric acid, diluted in the proportion of 1 part of concentrated acid with 1 part of water, by volume. Mix and set the flask aside for inversion 3 hours, provided the temperature is above 20° C., but longer if a lower temperature; if warmer than 25° C. two hours' standing is sufficient to complete the inversion. After inversion, dilute the solution to 100 cc., at the temperature of the direct polarization. Add a little zinc dust, in small quantities, and set aside for a few minutes. Filter and polarize in the jacketed tube with water circulation noting the temperature with a thermometer graduated to $\frac{1}{2}^{\circ}$ or $\frac{1}{3}^{\circ}$ C. So control the temperature that it shall be nearly the same as that of the direct polarization. Multiply the invert reading by 4, to ascertain I of the formula.

Calculation.—Use Steuerwald's Table No. 1. Select the constant (C), corresponding to the observed invert reading, interpolating to obtain closer approximations than in the table. Substitute the polarizations and constant in the following formula, in which t is the temperature of observation, D the direct, and I the invert polarizations:

$$\text{Sucrose by Clerget's method, } \frac{100(D+I)}{C-0.5t}.$$

D and I are always to be added arithmetically in cane sugar factory work.

Invert Polarization. Modification of the Herzfeld Method.—Make the direct polarization as has been described. Use a special 75/5/20-cc. inversion flask or a 100-cc. flask for the inversion. Using the special flask, measure 75 cc. of the delead solution in the flask and add 5 cc. of concentrated hydrochloric acid. Set aside at room temperature for 24 hours and then complete the volume to 100 cc. and proceed as in the previous method. If the ordinary flask is used, the delead solution should be measured into it with a 75 cc. pipette. Proceed with the polarization and calculations as in the previous method, except use Steuerwald's Table No. 2.

If the temperature of the laboratory is below 20° C. the inversion period should be lengthened or the flask be placed in a warmer room. In these analyses it is advisable to run a parallel test, letting the second flask stand two or three hours longer than the first. Accept the test in which complete inversion is indicated.

Glucose Determinations. Copper Solution.—Use Soxhlet's modification of Fehling's solution prepared with chemically pure reagents as follows: (A) Dissolve 69.28 grams of copper sulphate crystals in distilled water and dilute the solution to 1000 cc. (B) Dissolve 346 grams of tartrate of sodium and potassium (Rochelle salt), in about 600 cc. of distilled water; add 200 cc. of a solution of sodium hydroxide containing 516

¹ Errors are often traceable to measurements. A standardized pipette or burette should be used under the drainage conditions of its standardization or calibration.

STEUERWALD'S TABLE OF CONSTANTS, NO. 1.

(Inversion at Room Temperature with 30 cc. Acid.)

Inversion reading in the 200 mm. Tube.	Temperature at which the Observation is Made.					
	20° C.	22° C.	24° C.	26° C.	28° C.	30° C.
-25	143.51					
-24	143.45	143.43				
-23	143.39	143.38	143.36			
-22	143.34	143.32	143.30	143.29		
-21	143.28	143.26	143.24	143.23	143.21	143.20
-20	143.23	143.20	143.18	143.16	143.15	143.14
-19	143.17	143.15	143.12	143.10	143.09	143.07
-18	143.12	143.09	143.06	143.04	143.02	143.01
-17	143.06	143.03	143.00	142.98	142.96	142.94
-16	143.01	142.98	142.95	142.92	142.90	142.88
-15	142.95	142.92	142.89	142.86	142.83	142.81
-14	142.90	142.86	142.83	142.80	142.77	142.74
-13	142.84	142.80	142.77	142.74	142.71	142.68
-12	142.79	142.75	142.71	142.68	142.64	142.61
-11	142.73	142.69	142.65	142.61	142.58	142.55
-10	142.68	142.64	142.59	142.55	142.52	142.48
-9	142.62	142.58	142.53	142.49	142.45	142.42
-8	142.57	142.52	142.47	142.43	142.39	142.35
-7	142.51	142.46	142.41	142.37	142.33	142.29
-6	142.46	142.41	142.36	142.31	142.26	142.22
-5	142.40	142.35	142.30	142.25	142.20	142.15
-4	142.35	142.29	142.24	142.19	142.14	142.09
-3	142.29	142.24	142.18	142.13	142.07	142.02
-2	142.24	142.18	142.12	142.06	142.01	141.96
-1	142.18	142.12	142.06	142.00	141.94	141.88

grams of the hydroxide per 1000 cc., and then complete the volume to 1000 cc. with distilled water.

Solutions *A* and *B* must be kept in separate bottles. 25 cc. of each of these solutions are mixed and used in making the tests. The mixture should be made immediately before making the test.

Preparation of the Filtering Crucible.—The use of an alundum crucible or a fused silica Gooch crucible is optional. The alundum crucible should be used when laboratory conditions admit. It should be the $1\frac{3}{8} \times 1\frac{1}{2}$ inches (25 cc.) size, factory number, RA. 360. This number designates the porosity of the crucible.

The alundum crucibles when new must be treated as follows: Soak the crucibles thoroughly in nitric acid and follow this treatment with thorough washing, under filtering conditions, with a large quantity of very hot water. Follow the acid treatment and washing with soaking in hot Soxhlet's solution (mixed solutions *A* and *B*), and then wash the crucible as before with hot water. Reverse the crucible and repeat the washing with hot water. The reverse washing may be easily conducted as follows: Insert a single-hole No. 9 rubber stopper into a piece of glass tubing of corresponding size (the

STEUERWALD'S TABLE OF CONSTANTS, NO. 2.

(Herzfeld's Inversion Method.)

Reading in the 200 mm. Tube After Inversion = P	Temperature at which the Reading is made = t						
	32° C.	30° C.	28° C.	26° C.	24° C.	22° C.	20° C.
-18						145.51	145.54
-17				145.39	145.42	145.46	145.49
-16		145.27	145.30	145.34	145.37	145.41	145.44
-15	145.18	145.22	145.25	145.28	145.32	145.35	145.39
-14	145.12	145.16	145.19	145.23	145.26	145.30	145.34
-13	145.06	145.10	145.13	145.17	145.21	145.25	145.29
-12	145.00	145.04	145.08	145.12	145.16	145.20	145.24
-11	144.94	144.98	145.02	145.07	145.11	145.15	145.19
-10	144.88	144.93	144.97	145.01	145.06	145.10	145.14
-9	144.82	144.87	144.91	144.96	145.00	145.05	145.10
-8	144.77	144.81	144.86	144.90	144.95	145.00	145.05
-7	144.71	144.75	144.79	144.84	144.90	144.95	145.00
-6	144.65	144.70	144.74	144.79	144.84	144.89	144.95
-5	144.59	144.64	144.69	144.74	144.79	144.84	144.90
-4	144.53	144.58	144.63	144.68	144.74	144.79	144.85
-3	144.47	144.52	144.58	144.63	144.69	144.74	144.80
-2	144.41	144.46	144.52	144.58	144.63	144.69	144.75
-1	144.35	144.41	144.46	144.52	144.58	144.64	144.70

glass body of an oiler will serve) and about $1\frac{1}{2}$ inches long. Connect the stopper with the vacuum filtering flask. In washing the crucible, place it bottom up on the rubber stopper and apply suction while washing. Dry the washed crucible and then ignite it at a strong red heat during at least 30 minutes. The electric muffle furnace is well adapted for this heating, but in its absence a strong blast lamp flame is necessary. The prepared crucible should be kept in a desiccator until required for use.

After use in the collection and reduction of the copper oxide, the crucible must be soaked in nitric acid to dissolve out the copper. It must then be thoroughly washed by direct and reverse washing and be ignited as before. The walls of the crucible must be thoroughly wet with water before starting the filtration.

If the Gooch crucible is used, proceed as follows: Use a silica crucible provided with a flanged bottom and a platinum disk. Cover the disk, with suction, to a depth of about one-fourth inch with a felt of asbestos.

Short fiber filtering asbestos wool should be used. The wool should first be boiled with hydrochloric acid and then washed thoroughly with water by decantation. After the above treatment the asbestos wool should be digested with a hot Soxhlet's solution, diluted with an equal volume of water. This digestion should be followed with very thorough washing of the fiber with hot water. The prepared fiber should be suspended in water. The asbestos wool improves with use. The felt containing the copper should be accumulated in a bottle and from time to time the metal in it should be dissolved in nitric acid and the fiber be thoroughly washed with water, preparatory to reuse. Great care is necessary in using the Gooch crucible to avoid breaking the felt

and washing part of the fiber through with the filtrate. The filtrates should be examined and if necessary be refiltered.

The alundum crucibles shall be used with Spencer's funnel holder in which the joint is made at the top of the crucible. This holder may be of brass for glucose tests. Other types of alundum crucible holders may not be used. Serious errors have been traced to the use of such holders. The usual holders may be used with Gooch crucibles.

Preparation of the Sample and the Solution for Analysis.—The sample of molasses must be thoroughly mixed, that it may be uniform, with fine sugar crystals, if present, suspended throughout the mass. Slight warming of the molasses is necessary.

Immediately after mixing the sample proceed as follows in the preparation of the solution:

(a) For molasses containing more than 26 per cent of glucose: Weigh 5 grams of molasses, dissolve this in water and wash the solution into a 500-cc. flask and dilute to the mark. Do not add lead or other reagent. Mix the solution thoroughly by shaking, then add three teaspoonsful of kiesueighr (filtercel), and again shake thoroughly. Filter, returning the filtrate to the paper until it runs perfectly clear.

(b) For molasses containing more than 18 per cent and less than 26 per cent of glucose: Weigh 7.5 grams of the molasses and proceed as in (a).

(c) For molasses containing less than 18 per cent of glucose: Weigh 10 grams of molasses and proceed as in (a).

Proceed immediately with the analysis. The molasses solution contains no preservative and may alter perceptibly on standing.

Reduction of the Copper.—A supply of cold recently boiled distilled water must be provided, also very hot distilled water for washing the precipitate. Beakers of either 400-cc. or 500-cc. capacity and approximately 7 to 8 cm. diameter should be used. Always use the same size of beaker. These should preferably be of Jena, "Nonsol," Pyrex, or similar resistance glass. A glass or metal cover, like a watch-glass, having an opening in the center and fitted with a centigrade thermometer must be provided for the beakers.

Accurately measure 50 cc. of the filtrate (a), (b) or (c), as the case may be, into the 400- or 500-cc. beaker and add 25 cc. each of Soxhlet's copper solution (A) and alkali solution (B).

Cover the beaker, projecting the thermometer into the solution until it extends to within about 6 mm. of the bottom of the beaker. Place the beaker over the flame of a lamp and so adjust the heat that the solution will reach 100° C. in 4 minutes. When the temperature reaches 100° C., note the time and continue the heating for exactly 2 minutes, maintaining gentle ebullition. This interval should be timed with an accurate sand glass or an interval timer. At the conclusion of the boiling period, remove the lamp and the beaker cover and add 100 cc. of the cold, recently boiled, distilled water.

Filter off the cuprous oxide precipitate immediately, using the prepared alundum crucible (or Gooch). Wet the walls of the alundum crucible with water immediately before using it. Keep the level of the solution low in the crucible. A stirring rod ("policeman") should be used to guide the solution into the crucible and this should nearly touch the asbestos when beginning to pour into a Gooch crucible. The lip of beaker should be slightly greased with vaseline. Wash all of the cuprous oxide into the crucible with hot water and with the assistance of the rubber "policeman." Great care must be exercised that every part of the walls of the crucible be thoroughly washed.

Reduction to Metallic Copper.—*Votoček-Wedderburn Method.*)—Provide a small metal vessel, a copper beaker will answer. Place a pipe-stem triangle, bending down the wires to form legs, in the vessel to support the crucible. Arrange this so that the crucible will be held a few centimeters above the bottom of the vessel. Pour a little strong commercial alcohol into the beaker, cover it with a watch-glass and place it on a hot plate and heat it until the vapors of alcohol expel the air and condense on the under side of the cover. In the meantime, ignite the crucible, containing the cuprous oxide, to redness and maintain this temperature a short time to burn off organic matter. Allow the crucible to cool to faint redness, then place it in the beaker in the atmosphere of alcohol vapor and replace the cover. The reduction to metallic copper is almost instantaneous. Leave the crucible in the alcohol vapor until all danger of reoxidation has passed. If still warm when removed from the alcohol, the crucible should be placed in the desiccator preparatory to weighing. If cold, moisten the plating of copper and the walls of the crucible with pure alcohol and burn this with the crucible in an upright position, and then place the crucible in the desiccator.

Calculation of the Glucose.—Calculate the glucose by the Meissl-Hiller formula and table of factors as below. Rice's expanded Meissl-Hiller table may be conveniently used with samples containing 26 per cent or more glucose (5 grams molasses in 500 cc. i.e., 1:100) or 18 per cent and less, 2:100.

Let

Cu = milligrams of copper reduced;

P = polarization of the material;

W = weight of the material in 50 cc. of the solution;

F = factor from the table of Meissl and Hiller;

$Cu \div 2 = Z$ = the approximate absolute weight of glucose;

$100 Z \div W = y$, approximate per cent of glucose;

$100 P \div (P + y) = R$, relative number for sucrose;

$100 - R = I$, relative for number glucose;

$Cu \times F \div W$ = per cent glucose in the material.

The following examples illustrate the calculations and the use of the table: Molasses containing between 18 and 26 per cent glucose, (b) : Polarization (P) = 25; weight used 7.5 grams per 500 cc. or 0.75 gram per test; copper plating = 0.26 gram.

Substituting in the formula,

$$Cu \div 2 = Z = 0.13;$$

$$100 Z \div W = y = 13 \div 0.75 = 17.33;$$

$$100 P \div (P + y) = R = 2500 \div 42.33 = 59 \text{ (Use nearest whole number);}$$

$$100 - R = I = 100 - 59 = 41 \text{ (Use nearest whole number);}$$

$$R : I = 59 : 41.$$

Under "approximate absolute weight of glucose, Z " in the table and opposite the nearest ratio to 59 : 41, find the factor 53.2, then

$Cu \times F \div W$ = per cent glucose = $0.26 \times 53.2 \div 0.75 = 18.44$, per cent glucose in the molasses.

Rice's expanded Meissl-Hiller table¹ may be used for molasses containing more than 26 per cent or less than 18 per cent of glucose: *Example:* Polarization = 26; weight used (c) 10 grams per 500 cc. or 2 grams per 100 cc.; copper plating = 0.25 gram.

¹ Proceedings Eighth International Congress App. Chem.; "Hand Book for Cane Sugar Manufacturers," 5th Ed., Spencer, p. 485.

MEISSL AND HILLER'S FACTORS

FOR THE DETERMINATION OF MORE THAN 1 PER CENT OF INVERT-SUGAR

Ratio of Sucrose to Invert-sugar= $R : I$	Approximate Absolute Weight of Invert-sugar = Z .						
	Milligrams.						
	200	175	150	125	100	75	50
	Per Cent	Per Cent	Per Cent	Per Cent	Per Cent	Per Cent	Per Cent
0 : 10	56.4	55.4	54.5	53.8	53.2	53.0	53.0
10 : 90	56.3	55.3	54.4	53.8	53.2	52.9	52.9
20 : 80	56.2	55.2	54.3	53.7	53.2	52.7	52.7
30 : 70	56.1	55.1	54.2	53.7	53.2	52.6	52.6
40 : 60	55.9	55.0	54.1	53.6	53.1	52.5	52.4
50 : 50	55.7	54.9	54.0	53.5	53.1	52.3	52.2
60 : 40	55.6	54.7	53.8	53.2	52.8	52.1	51.9
70 : 30	55.5	54.5	53.5	52.9	52.5	51.9	51.6
80 : 20	55.4	54.3	53.3	52.7	52.2	51.7	51.3
90 : 10	54.6	53.6	53.1	52.6	52.1	51.6	51.2
91 : 9	54.1	53.6	52.6	52.1	51.6	51.2	50.7
92 : 8	53.6	53.1	52.1	51.6	51.2	50.7	50.3
93 : 7	53.6	53.1	52.1	51.2	50.7	50.3	49.8
94 : 6	53.1	52.6	51.6	50.7	50.3	49.8	48.9
95 : 5	52.6	52.1	51.2	50.3	49.4	48.9	48.5
96 : 4	52.1	51.2	50.7	49.8	48.9	47.7	46.9
97 : 3	50.7	50.3	49.8	48.9	47.7	46.2	45.1
98 : 2	49.9	48.9	48.5	47.3	45.8	43.3	40.0
99 : 1	47.7	47.3	46.5	45.1	43.3	41.2	38.1

Opposite 0.25 gram copper (nearest number 0.2497), in the 2-gram column and under 30°, the nearest number to the polarization of the molasses, find 13.22, the per cent glucose in the sample. Rice's table may readily be extended to include other percentages of glucose.

56. Sugars.—*Moisture.*—Heat 5 g ams of sugar, distributed over the bottom of shallow dish at 105° C. if the sugar be the customary large hard crystal, for 3 hours. Cool in a desiccator, then weigh. Return the dish to the oven and heat for a further period of one-half hour. If the loss of weight during the second period is inappreciable, consider the sugar to be dry and calculate the percentage of loss as moisture. The alternative method of a single drying period of three hours and a half is permissible. After heating, during three and a half hours at 105° C., the sugar is to be considered dry. If the sugar is of soft small crystal and low test the temperature of drying should be 100° C. At least one composite sample of sugar, representing 24 hours work, shall be dried daily. If there is a tendency for the sugars to contain 1 per cent or more of moisture at least four control samples, representing the four watches, shall be dried daily.

Ash.—See Ash Determinations (58).

Direct Polarization.—Wash one normal weight of sugar, from a nickel weighing capsule, with water into a narrow-necked graduated flask. A nickel funnel may be used in transferring the sugar or more conveniently a Stiff flask. The sugar in the capsule

should be moistened with water and let stand a moment to soften before transferring. Together with the washings, sufficient water should be added to fill the flask about four-fifths full. Dissolve the crystals by imparting a rotary motion to the flask. Examine the solution, by holding the flask above the level of the eye, to verify the complete solution of the sugar. Add approximately 2 cc. of 54.3° Brix subacetate of lead solution, varying the quantity according to the grade of the sugar, and always using the minimum quantity that insures a good clarification. Mix the contents of the flask, add about 5 cc. of alumina cream and finally dilute the solution to 100 cc., breaking foam with a drop of ether. Mix the contents of the flask very thoroughly by vigorous shaking and pour upon a filter, covering the funnel with a watch-glass. The entire contents of the flask must be poured at once upon the filter. Reject about 25 cc. of the first running of the filtrate, which displaces the moisture of the paper. If the subsequent filtrate be cloudy, reject the solution and prepare another, increasing or decreasing the quantity of lead solution as may be necessary. Polarize the filtrate, using the 200 mm. observation tube. If a Clerget test is to be made, use the jacketed tube (Landolt), and note the temperature of the direct observation.

Invert Polarization (Clerget).—Delead a part of the filtrate from the direct polarization by the addition of sodium oxalate. Add the oxalate little by little until no very perceptible precipitate forms with it. Follow the oxalate with a few milligrams (estimated) of dry kieselguhr (filtercel), mix thoroughly and then filter. Complete deleading is neither necessary nor desirable. Accurately measure 50 cc. of the delead solution into a 100-cc. flask, add 25 cc. of water and then proceed with the inversion, observations and calculations by the modified Herzfeld's method as described for molasses. Or, invert 50 cc. of the solution with 30 cc. acid, as described in Steuerwald's method for molasses and use Table No. 1 for the calculations.

Great care must be exercised at all stages of the sugar sampling and analysis. The samples must be rapidly mixed on a glass or metal plate, and then be subsampled into a glass jar. Obviously the plate must not be cooler than laboratory temperature. Samples for run reports may be composited during a fortnight, but this period shall not be extended. Experiments have shown that samples retain their polarization during this period, but that those held for a much longer time sometimes deteriorate.

Only the most accurate precision glassware should be used in sugar testing. The polariscope should be checked with standard quartz plates and these plates should not have been wiped within several hours of their use.

Temperature Corrections.—All polarizations of sugars shall be corrected to the standard temperature, 20° C., by Browne's formulæ (Handbook of Sugar Analysis, p. 260). For sugars polarizing 96° or above use formula No. 1 and for those of lower polarization use formula No. 2. In these formulæ P^{20} is the corrected polarization, P^t the observed polarization and t is the temperature of the observation:

$$\text{No. 1. } P^{20} = P^t [1 + 0.0003 (t - 20)].$$

$$\text{No. 2. } P^{20} = P^t + 0.0015 (P^t - 80) (t - 20).$$

These formulæ are for use pending further instructions.

Glucose.—The glucose shall be determined in sugars by the method described for molasses, preparing the solution by filtration with kieselguhr and without the use of either the normal or basic lead acetate. The solution strength should be such as to yield approximately 220 to 280 milligrams of copper in the reduction process. Rice's

table may be conveniently used in estimating the solution strength. For example, it may be noted by the table that 20 grams of a sugar containing 1.2 per cent of glucose, dissolved in 100 cc. will yield 257.6 milligrams of copper in the reduction. Therefore this solution strength should be used for sugars containing from about 1 to 1.33 per cent of glucose.

The calculations may be made with the assistance of Rice's table, if within its range, or by the Meissl-Hiller table. Experiments made in the Central Control Laboratory indicate that it is advisable to vary the solution strength so as to keep the weight of reduced copper within somewhat narrow limits.

57. Condensation and Waste Waters.—Frequent sucrose tests shall be made in water from the condensers and tail-pipes. It rarely occurs that the water contains sufficient sucrose for a polariscopic test. The sucrose content is usually very small and may be estimated with sufficient accuracy by the α -naphthol test. This test is made as follows: Add 5 drops of a 20 per cent solution of α -naphthol in alcohol to 2 cc. of the water in a test-tube, then add 10 cc. of concentrated sulphuric acid, using a pipette to conduct the acid to the bottom of the tube. If sucrose is present a violet zone forms between the two liquids; in the presence of 0.1 per cent of sucrose the reaction is obscured by the darkening of the solution; with 0.01 per cent sucrose the color zone is that of a very dark-red wine; with 0.001 per cent sucrose the entire solution is colored. Impure water will sometimes impart a faint color in the above test in the absence of sucrose, but this is usually readily distinguished from the color produced by sugar.

If condenser water contains a distinct trace of sugar, the percentage shall be determined by the analysis (chemical method), of the concentrate obtained in evaporating a liter or more of the water. The sugar loss shall be estimated from the calculated weight of the condenser water.

A very thorough control of the boiler-feed water shall be made by means of the α -naphthol test and very frequent samples. If sugar is discovered in the water the engineer on duty must be immediately advised and tests of the water from each tail-pipe must be made to locate the source of the sugar. If the boiler-feed water contains no sugar, it is unnecessary to test that from the tail-pipes.

58. Special Methods of Analysis.—Ash Determinations.—The sulphated-ash method is to be used in all ash determinations. In this method the material contained in a shallow silica dish is saturated with pure sulphuric acid, then gently heated until intumescence ceases. The heat is now to be gradually increased and the carbon burned. An electric muffle heated to dull redness is the best device for ashing sugar products. Care must be exercised not to fuse the ash. The sulphuric acid must be in sufficient quantity to convert the mineral constituents of the material into sulphates. To reduce the sulphated ash to terms of the normal ash, deduct one-tenth of its weight.

The following approximate quantities of the sugar products and sulphuric acid should be used in each test: 2 grams sugar and 0.5 cc. acid; 10 grams juice and 1 cc. acid; 2 grams molasses and 1 cc. of acid.

Methods of Drying.—Methods for bagasse, filter cake and hard sugars are given with the methods for these materials.

The drying of sugar-house products is not a simple problem, and it is difficult to determine the proper moment at which to discontinue the operation.

Juice, filter-cake and molasses should be dried to constant weight in a vacuum oven. This also applies to soft sugars, though these are usually dried in a water oven. A temperature of 70° and a vacuum of 25 inches is recommended. Shallow dishes should be

used. The lead-tin caps for bottles, 3-inch size, are well adapted for this purpose. In the absence of a vacuum oven the analysis must be conducted in an ordinary oven, the temperature varying with the material, but should not exceed 100°C . Decomposition of many materials occurs in the ordinary oven, so considerable judgment and experience are required to determine the proper length of the heating period of low products. Vacuum ovens that are heated by steam may conduct the drying at a higher temperature than the thermometer in the oven indicates owing to the conductivity of the shelves. The shelves or dishes should rest upon insulating material. Small vacuum-distilling apparatus, consisting of a flanged porcelain vessel and glass dome, connected with the vacuum system and heated by boiling water, gives good results. The dishes must rest upon the bottom of the porcelain vessel.

The expression "to constant weight" should not be construed too literally. This would sometimes prolong the drying period very excessively and then without increased accuracy of determination. With certain materials there is a loss of weight in drying due to decomposition. It is often advisable, for example, for molasses if dried in an ordinary oven, to heat about 2 grams of the sample, distributed over the bottom of a shallow dish, 10 hours at 100°C . and at the end of this period consider the sample to be dry. A more dependable result is obtained by heating the sample about 8 hours at low temperature, 60° to 70°C ., and then continuing the heating at 100°C . until the loss of weight between weighing intervals is but slight. The material should be dissolved in a little distilled water, to facilitate its distribution over the dish.

ESTIMATION OF THE PRODUCTS IN PROCESS OF MANUFACTURE

59. Stock-taking.—The permanent records of the laboratory should include the dimensions and capacities of all tanks, pans, crystallizers, mixers, etc., in the factory. Each piece of apparatus should bear a suitable mark for identification.

At the close of a Run the chemist, accompanied by assistants, should rapidly pass through the factory and measure and sample all material in process. At the same time the head pan-boiler should indicate with chalk the depth of massecuite in each pan and draw proof-stick samples. Beginning two or three days before the close of a Run, the head pan man should record the "inches out" for each crystallizer as it is filled.

Should a suspension of milling or other condition permit, instead of measuring the material in the pans, the strikes may be concluded and purged, using syrup and molasses set aside for this purpose. Account must be made of such syrup and molasses. The massecuite produced may be measured or it may be purged and account be taken of the sugar and molasses.

In measuring the juice and syrup in the tanks it is usually easier to measure the depth of the empty space from the top of the tank, or, in other words, the "wantage" or "inches out" rather than the depth of the liquor. The stock notes should always distinctly indicate the quantity of material in the vessel, and if empty, that such is the case. There must be an entry for each container.

Samples should be drawn, representing each class of material in stock. The laboratory records will show the analyses of the massecuites, so these need not be sampled.

From these data the quantity of materials of all kinds in stock at a given hour may readily be calculated. All stock data and calculations should be entered in a book to facilitate the work of estimating the sugar and molasses. It is preferable that all the calculations be made in the book itself. Full precautions against error shall be observed

in stock-taking and sampling. The figures shall be accepted without deductions. Personal error and judgment must be eliminated in so far as may be possible. The superintendent is expected to require full and explicit entries on the stock sheets. Careless writing shall not be permitted.

It is sometimes more convenient to make the stock estimates a little in advance or after the close of the Run, in order to take advantage of a shut-down. In the one case the cane ground from the hour of stock-taking to the end of the Run is carried as "stock," and the sugar it will produce is estimated on a basis of the yield for the rest of the period. In the other case the estimated yield from the cane ground from the end of the Run to the shut-down is to be deducted from the total.

Estimation of the Sugar in Process.—The sugar in process in the raw juice may be estimated on a basis of the "sucrose retained" in a previous Run, or, if such data are not at hand, the "retention" may be estimated with the aid of a commercial sugar formula allowing for the losses usually experienced by the factory. As the quantity of raw juice in stock is small, an over or under estimate here will introduce a very small error. Likewise the yield from clarified juice may be estimated at a somewhat higher rate, and that from syrup at a still higher rate, as these materials have already undergone a part of the manufacturing process. When first massecuite is made without boiling in molasses, its sugar yield may be estimated upon the above basis. It is, however, usually more convenient to estimate the sugar and molasses in process by means of the formulæ and a weighted average analysis of all the material, including juice, syrup, molasses, etc.

When molasses is "boiled-in," the yield of sugar from the massecuites may be estimated by means of one of the following formulæ:

- (1) Let x = percentage yield of commercial sugar from the massecuite;
 B = degree Brix of the massecuite, or weight of solids;
 P = polarization of the massecuite, or weight of sucrose;
 p = polarization of the commercial sugar;
 S = percentage of solids in the commercial sugar;
 M = coefficient of purity of the final molasses.

Then

$$x = \frac{100 P - BM}{p - SM/100}$$

- (2) Let x = percentage yield of water-free commercial sugar in terms of the dry matter (Brix) of the massecuite, molasses, etc.;

100 = weight of the massecuite, molasses, etc.;
 P = coefficient of purity of the massecuite, etc.;
 P' = coefficient of purity of the commercial sugar;
 B = Brix or dry matter of the massecuite, etc.;
 M = coefficient of purity of the final or residual molasses.

Then

$$x = 100 \frac{P - M}{P' - M}$$

This method of calculation gives the yield of sugar with considerable accuracy. The results would be accurate if true Brix, polarizations, and purities could be used,

provided no solids are removed in the processes of manufacture other than in the products.

It will be noted in these methods that the yield includes all of the sugar presumed to be available, as of one grade. If more classes of sugar than one are made, their relative proportions must be ascertained from the experience of the factory and calculated yield be apportioned accordingly, figuring each class to the average polarization of that grade of sugar.

Estimation of the Molasses in Process.—The molasses in process is estimated from the total solids (Brix) and the sucrose remaining after the deduction of these in the products.

The weight of solids (Brix) in materials in process of manufacture minus the solids in the sugar that it is estimated these will produce, leaves the solids in the molasses in process. This number divided by the degree Brix assumed for the molasses in process, pointing off for percentage, gives the weight of molasses. The sucrose is calculated from the degree Brix and the assumed purity. It is not usual to allow for the air occluded by the molasses in estimating the volume, but to divide the weight of molasses by the weight of the gallon (air free) as deduced from the degree Brix. This may result in an important error and the apparent reduction or even disappearance of the "undetermined" or "loss" number in the sucrose balance. If the measurement of the molasses is used in the calculations an arbitrary correction, based upon experience, should be made.

The molasses flowing from the storage tank into the tank cars carries more or less air with it, according to the position of the discharge pipe and the density of the molasses, hence the tank-car measurement is usually slightly larger than that in the tank. The measurement in the car, however, is the one to be used in reporting the yield of molasses whenever possible, as it is the commercial number. So far as the sucrose control of the factory is concerned, this is provided for if the molasses is weighed in the cars. Unfortunately railway track arrangements often preclude weighing the molasses.

Run Calculations.—The following example illustrates a few of the run calculations and is based upon the stock data in the tabulation, page 30:

Juice.—Since the temperature of measurement is above the standard for the hydrometers ($17\frac{1}{2}^{\circ}\text{C.}$), a volume correction must be applied. Such corrections are always approximations on account of the wide difference between the temperature of measurement and the standard, also for other reasons. Gerlach's table of corrections for Brix hydrometers may be used or a table showing the volume of sugar solutions at various temperatures. The former table is the more convenient.

Brix of the juice at $17\frac{1}{2}^{\circ}\text{C.}$	14.0
Correction (from Table) at 75°C.	5.9
Brix at 75°C.	9.1

The weight of a cubic foot of this juice at 75°C. is 64.62 lbs. avoirdupois as is given in the table, page 35.

$$\begin{aligned}
 \text{Weight of juice} &= 4,221 \times 64.62 = 272,761 \text{ lbs. avo.} \\
 \text{" solids} &= 272,761 \times .14 = 38,186 \text{ " " } \\
 \text{" sucrose} &= 272,761 \times .1204 = 32,840 \text{ " " }
 \end{aligned}$$

Material.	Measurement Temp. C°.	Quantity.	Weight, Lbs. Sp.	Corrected Brix or Solids.	Per cent Sucrose.	Purity.
Juice: Raw	None	in stock.				
“ Cachaza.....						
“ Defecated.....						
Syrup.....	75	4,221 cu.ft.		14.0	12.04	
Molasses.....	70	2,257 “		56.5	50	
1st Masecuite.....	60	5,067 “		78.54	43.53	
2d Masecuite.....	60	2,772 “		92.28	80.28	
Final molasses, yard tank...	60	6,780 “		95	57.95	
Final molasses, shipped, run		82,143 gals.		88.5	27.88	31.5
Final molasses, shipped, to		384,514 “	4,537,265	88.5	28.79	32.53
previous date.....		440,000 “	5,192,030	88	28.16	32
Final molasses, made and es-		442,885 “	5,226,050			
imated to previous date...						
Assumed analysis of mo-				88	27.28	31
lasses, in process.....						
Sugar, made this run, bags of						
320 lbs. Sp.....		72,400 bags		99.2	*96.6	
Sugar, to previous date, bags						
of 320 lbs. Sp.....		65,481 “				
Sugar, made and estimated						
to previous date, bags of						
320 lbs. Sp.....		63,500 “				

* The polarization of the sugar, corrected for temperature error, shall be used in all technical reports of the Company. See page 25.

Syrup.—Calculating as for juice: Brix at 70° C.=56.5—4.9=51.6 and the corresponding weight per cubic foot is 77.42 lbs. avoird.

$$\begin{aligned}
 \text{Weight of syrup} &= 2,257 \times 77.42 = 174,737 \text{ lbs. avoird.} \\
 \text{“ solids} &= 174,737 \times .565 = 98,726 \text{ “} \\
 \text{“ sucrose} &= 174,737 \times .50 = 87,368 \text{ “}
 \end{aligned}$$

Molasses.—Calculating as for juice: Brix of the molasses at 60° C.=78.54—3.41 (limit of table)=75.13 and the corresponding weight per cubic foot is 86.26 lbs. avoird.

$$\begin{aligned}
 \text{Weight of molasses} &= 5,067 \times 86.26 = 437,079 \text{ lbs. avoird.} \\
 \text{“ solids} &= 437,079 \times .7854 = 343,282 \text{ “} \\
 \text{“ sucrose} &= 437,079 \times .4353 = 190,260 \text{ “}
 \end{aligned}$$

First Masecuite.—The estimation of the weight per cubic foot is calculated as for juice, etc., but is a rougher approximation: The Brix at 60° C.=92.28—3.40 (limit of the table)=88.9, and the corresponding weight per cubic foot is 92.54 lbs. avoird.

$$\begin{aligned}
 \text{Weight of first masecuite} &= 2,772 \times 92.54 = 256,521 \text{ lbs. avoird.} \\
 \text{“ solids} &= 256,521 \times .9228 = 236,718 \text{ “} \\
 \text{“ sucrose} &= 256,521 \times .8028 = 205,935 \text{ “}
 \end{aligned}$$

Second Masecuite.—Proceed as for first masecuite: The Brix at 60° C. is 91.6 and the corresponding weight per cubic foot is 93.29 lbs. avoirdupois.

$$\begin{aligned}\text{Weight of second masecuite} &= 6,780 \times 93.29 = 632,506 \text{ lbs. avoirdupois.} \\ \text{" solids} &= 632,506 \times .95 = 600,881 \text{ " } \\ \text{" sucrose} &= 632,506 \times .5795 = 366,537 \text{ " }\end{aligned}$$

Final Molasses (Yard tank).—The weight of a unit volume of molasses (air-free), may be deduced from the analysis, but an arbitrary allowance must be made in estimating the actual weight in stock. This allowance must be based upon the experience of the factory. An air-free molasses that weighs, *e.g.*, 12 lbs. per gallon, may only weigh per actual measured gallon 11 lbs. or less. The weight varies with the container (storage tank or tank-car) and the length of time in storage. For the purposes of this example we will estimate the weight at 11 lbs. Spanish though the air-free weight would be 12.11 lbs. Sp. Shipping weights should when possible be used in these calculations. These weights are usually in Spanish libras, hence, this system is used in this part of the estimates.

$$\begin{aligned}\text{Weight of molasses} &= 82,143 \times 11 = 903,573 \text{ lbs. Sp.} \\ \text{" solids} &= 903,573 \times .885 = 799,662 \text{ " } \\ \text{" sucrose} &= 903,573 \times .2788 = 251,916 \text{ " }\end{aligned}$$

The figures obtained in the above calculations should be tabulated as follows:

Stock Resumé

Materials in Process.	Weight.	Solids.	Sucrose.
	Lbs. Avoirdupois.	Lbs. Avoirdupois.	Lbs. Avoirdupois.
Juice.....	272,761	38,186	32,840
Syrup.....	174,737	98,726	87,368
Molasses.....	437,079	343,282	190,260
1st Masecuite.....	256,521	236,718	205,935
2d Masecuite.....	632,506	600,881	366,537
Totals.....	1,773,604	1,317,793	882,940

By formula (1), page 28, we have

$$x = \frac{100P - BM}{p - SM/100} = \frac{100 \times 882,940 - 1,317,793 \times 31}{96.6 - 99.2 \times 31/100} = 720,480 \text{ lbs. avoirdupois. of commercial}$$

sugar in process of 96.6 polarization and 99.2 solids, as per the average analysis for the Run. The number 31 is the assumed purity of the molasses in process. The dry matter (Brix) of the sugar is $720,480 \times 0.992 = 714,719$ lbs. avoirdupois.

The weight of sugar should be converted into Spanish libras by dividing by the equivalence of the Spanish libra (see page 10), 1.0143 lb. avoirdupois; $720,480 \div 1.0143 = 710,325$ libras. The weight of a bag of sugar was formerly 13 arrobas or 325 libras, but owing to difficulty in obtaining full-size bags (48" x 29" English), the Company has adopted 320 libras as the capacity of a bag. The sugar in process is, therefore, $710,325 \div 320 = 2219$ bags.

The weight of molasses in process is deduced from the weights of dry matter in process and that in the sugar: $1,317,793 - 714,719 = 603,074$ lbs. dry matter (Brix), in the molasses in process. Since the molasses is assumed to be of 88° Brix, its weight is $603,074 \div 0.88 = 685,311$ lbs. It is now convenient to convert this number into Spanish libras or the weight of a gallon into libras. Choosing the former we have $685,311 \div 1.0143 = 675,649$ libras. We have assumed the gallon to weigh 11 libras, $\therefore 675,649 \div 11 = 61,422$ gallons, in process.

Résumé of Yields:

Sugar made this Run	72,400	bags	
“ “ to previous date.....	65,481	“	
“ in process.....	2,219	“	
Made and estimated to date.....	140,100	“	
“ “ “ “ previous date....	63,500	“	
“ “ “ “ this Run.....	76,600	“	
Molasses in yard tank	82,143	gals.	903,573 lbs. Sp.
“ shipped this run.....	384,514	“	4,537,265 “
“ “ to previous date.....	440,000	“	5,192,000 “
” in process.....	61,422	“	675,649 “
Made and estimated to date.....	968,079	“	11,308,487 “
“ “ “ “ previous date....	442,885	“	5,226,055 “
“ “ “ this Run.....	525,194	“	6,082,432 “

The calculations may be varied somewhat by calculating the average purity of the materials and using the second formula, page 28, or by converting to Spanish libras at an earlier stage, etc., but the calculations given illustrate the general method to be followed.

MISCELLANEOUS CALCULATIONS

60. Saturation.—The saturation water should be weighed where suitable arrangements can be made for tanks and scales. (*See 35.*) If the analyses could give true, instead of apparent numbers, and if the juice were of uniform composition throughout the stalks of cane, the saturation could be deduced by an inferential method from the laboratory data.

The water is sometimes measured at temperatures ranging from so low as 20° C. to often 85° C. The temperature is usually between 50° and 70° C. owing to the use of return water from the evaporators, etc. The following table gives the weight per cubic foot of water within this range. If a wider range is required, refer to the full table:¹

¹ Calculated by U. S. Bureau of Standards except as regards libras. ‘Handbook for Cane Sugar Manufacturers,’ 5th Ed. Spencer, p. 417.

WEIGHT OF 1 CUBIC FOOT OF WATER.

POUNDS AVOIRDUPOIS AND SPANISH LIBRAS.

Temp. Deg. C.	1 Cubic Foot.		Temp. Deg. C.	1 Cubic Foot.	
	Lbs. Av.	Libras Sp.		Lbs. Av.	Libras Sp.
50	61.615	60.746	60	61.314	60.448
51	.587	.718	61	.281	.416
52	.558	.690	62	.249	.384
53	.529	.661	63	.216	.352
54	.499	.632	64	.182	.318
55	.469	.602	65	.148	.285
56	.439	.573	66	.115	.253
57	.410	.544	67	.080	.218
58	.377	.511	68	.046	.184
59	.346	.480	69	.010	.149

The average temperature of measurement of the water should be ascertained and the corresponding weight per cubic foot be taken from the table.

The saturation should be reported in percentage terms of the weight of the cane.

It may occur that the actual weight or volume of the water cannot be ascertained, through accident to a meter or otherwise. In this event it is necessary to estimate the saturation number from the dilution, taking account of both the efficiency of the milling and that of the saturation water. When saturating moderately and obtaining about 90 per cent extraction, the dilution number is approximately 75 per cent of the saturation number. With large saturation and an extraction of 95 per cent the dilution number approximates 90 per cent of the saturation number. Such estimations are very unsatisfactory and introduce inaccuracies in the calculation of the extraction number and the bagasse and cane analyses.

61. Dilution.—The dilution of the juice (6) is calculated from the degrees Brix of the crusher and mixed juices and shall be expressed in percentage terms of the cane.

It is customary to reduce the degree Brix of the crusher juice to a basis of that of the mixed juices, obtained from full train of mills in dry crushing, by a factor deduced from experimental data. Sample the crusher juice and the mixed juices from the entire train of mills and also separately sample the crusher juice while operating without saturation. Brix each sample. Factor = degree Brix of mixed juices ÷ degree Brix of crusher juice.

Multiply the degree Brix of the crusher sample by the factor to ascertain the Brix of the so-called normal juice.

Let B and b represent respectively the degrees Brix of the normal and the diluted or mixed juice and x the percentage of dilution, then

(1) $x = 100(B - b)/B = 100 - 100b/B$, the dilution per cent diluted juice;

(2) $x = 100(B - b)b = 100B/b - 100$, the dilution per cent normal juice.

Multiply the value of x by the corresponding dilute juice or normal juice extraction and point off for percentage to reduce the dilution numbers to terms of the cane. Formula (2) is not used by this Company except in comparisons.

62. Mill Extraction.—The term “mill extraction” is to be interpreted as “the percentage of the sucrose in the cane that is extracted in the juice,” also the “normal juice (calculated) extracted per cent cane.” Both statements of mill extraction are used in the Company’s reports.

(1) Extraction, sucrose per cent sucrose in the cane = $\text{weight of sucrose in the mixed juice} \div (\text{weight of sucrose in the mixed juice} + \text{weight of sucrose in the bagasse}) \times 100$.

(2) Extraction, normal juice per cent cane = $\text{weight of normal juice (calculated)} \div \text{weight of cane} \times 100$.

In the event of an accident to the juice scales, the weight of the juice must be deduced from its measured volume. In the absence of accurate coefficients of expansion, the weight may be calculated as is shown in the following example: Measured volume of juice at 30° = 100,000 cubic feet; degree Brix at 17.5° C. = 15.0.

Degree Brix at 17.5° C.	= 15.0
Temperature correction (Gerlach’s table) at 30° C.	= .87
Apparent degree Brix at 27°	= 14.13

The weight (avoirdupois) of a cubic foot of juice at 14.13° Brix, from the table in the back of this pamphlet, is 65.95 lbs., ∴ 100,000 cubic feet of juice weigh 6,595,000 lbs.

The error in this method of figuring the weight is well within the limits of error of the measurements.

63. Bagasse.—When saturation is employed the weight of bagasse is calculated as follows: (Weight of cane + weight of saturation water) – weight of the mixed juice = weight of bagasse. There are two sources of error in this method of estimating the weight of the bagasse, always assuming weights and measures to be correct: (1) The evaporation of moisture from the bagasse in its passage through the mills. This error could be reduced by an arbitrary estimate of the loss of moisture but cannot be eliminated. For the present, no correction is to be made for this loss. (2) Leakage of water from the mill journals with older types of mills, is a source of error. This error can usually be obviated.

64. Available Sugar and Efficiency Number.—A part of the formula of Geerligs-Winter-Carp shall be used with the factory’s efficiency number in calculating the available sugar, as follows: Theoretical retention number = $100 (1.4 - 40/P)$, in which P is the purity coefficient of the raw juice; actual retention number $\div$ theoretical number $\times 100$ = Efficiency Number. The theoretical retention number applied to the sucrose per cent cane extracted by the mills, in the juice, gives the yield of sucrose (uncorrected), per cent cane. This number multiplied by the efficiency number gives the probable actual yield of sucrose. The yield of sucrose is calculated to terms of 96° sugar by dividing by 0.96. These calculations are simplified by the use of the tables¹ supplied the laboratories by the Company.

¹ See page 482, “Handbook for Cane-Sugar Manufacturers.” Spencer.

65. Table Showing the Weight per Cubic Foot and U. S. Gallon (231 Cu.In.) and Solids (Brix) of Sugar Solutions at 17½° C.

(Based upon Stammer's Table)

Degree Brix	Weight of		Solid (Brix) per		Degree Brix	Weight of		Solids (Brix) per	
	1 cu.ft.	1 gal.	1 cu.ft.	1 gal.		1 cu.ft.	1 gal.	1 cu.ft.	1 gal.
	Lbs.	Lbs.	Lbs.	Lbs.		Lbs.	Lbs.	Lbs.	Lbs.
1.0	62.59	8.36	0.63	0.08	17.50	66.85	8.93	11.70	1.57
.5	62.72	8.38	0.94	.08	.75	66.92	8.94	11.88	1.59
2.0	62.84	8.39	1.26	.17	18.0	66.99	8.95	12.06	1.61
.5	62.96	8.40	1.57	.21	.25	67.06	8.96	12.24	1.64
3.0	63.08	8.42	1.89	.25	.50	67.13	8.97	12.42	1.66
.5	63.20	8.44	2.21	.30	.75	67.20	8.98	12.60	1.69
4.0	63.32	8.46	2.53	.34	19.0	67.27	8.99	12.78	1.71
.5	63.44	8.48	2.86	.38	.25	67.34	9.00	12.96	1.74
5.0	63.57	8.50	3.18	.43	.50	67.41	9.01	13.14	1.76
.5	63.70	8.52	3.50	.47	.75	67.48	9.02	13.32	1.79
6.0	63.83	8.53	3.83	.51	20.0	67.55	9.03	13.51	1.81
.5	63.95	8.55	4.16	.56	.25	67.62	9.04	13.69	1.83
7.0	64.08	8.57	4.49	.60	.50	67.69	9.04	13.88	1.85
.5	64.21	8.59	4.82	.64	.75	67.76	9.05	14.06	1.88
8.0	64.34	8.60	5.15	.69	21.0	67.83	9.06	14.24	1.90
.5	64.47	8.61	5.48	.73	.25	67.90	9.07	14.42	1.93
9.0	64.60	8.63	5.81	.78	.50	67.97	9.08	14.61	1.95
.5	64.72	8.65	6.15	.82	.75	68.04	9.09	14.79	1.98
10.0	64.84	8.67	6.48	.87	22.0	68.11	9.10	14.98	2.00
.25	64.91	8.68	6.65	.89	.25	68.18	9.11	15.17	2.03
.50	64.97	8.69	6.82	.91	.50	68.25	9.12	15.36	2.05
.75	65.04	8.70	6.99	.94	.75	68.32	9.13	15.54	2.08
11.0	65.11	8.71	7.16	.96	23.0	68.39	9.14	15.73	2.10
.25	65.18	8.71	7.33	.98	.25	68.46	9.15	15.92	2.13
.50	65.24	8.72	7.50	1.00	.50	68.54	9.16	16.11	2.15
.75	65.31	8.73	7.67	1.03	.75	68.61	9.17	16.29	2.18
12.0	65.37	8.74	7.84	1.05	24.0	68.68	9.18	16.48	2.20
.25	65.44	8.75	8.01	1.08	.25	68.75	9.19	16.66	2.23
.50	65.51	8.76	8.19	1.10	.50	68.82	9.20	16.86	2.25
.75	65.58	8.77	8.36	1.12	.75	68.89	9.21	17.05	2.28
13.0	65.64	8.78	8.53	1.14	25.0	68.96	9.22	17.24	2.31
.25	65.71	8.78	8.70	1.17	.25	69.04	9.23	17.43	2.33
.50	65.77	8.79	8.88	1.19	.50	69.11	9.24	17.62	2.36
.75	65.84	8.80	9.05	1.21	.75	69.19	9.25	17.81	2.38
14.0	65.91	8.81	9.23	1.23	26.0	69.26	9.26	18.01	2.41
.25	65.98	8.81	9.40	1.26	.25	69.34	9.26	18.20	2.44
.50	66.04	8.82	9.58	1.28	.50	69.41	9.27	18.39	2.47
.75	66.11	8.83	9.75	1.31	.75	69.48	9.28	18.58	2.49
15.0	66.18	8.84	9.93	1.33	27.0	69.55	9.29	18.78	2.51
.25	66.25	8.85	10.10	1.36	.25	69.62	9.30	18.98	2.53
.50	66.31	8.86	10.28	1.38	.50	69.69	9.31	19.17	2.56
.75	66.38	8.87	10.45	1.40	.75	69.76	9.32	19.36	2.59
16.0	66.44	8.88	10.63	1.42	28.0	69.84	9.33	19.56	2.61
.25	66.51	8.89	10.80	1.44	.25	69.91	9.34	19.75	2.63
.50	66.58	8.90	10.97	1.47	.50	69.99	9.35	19.95	2.66
.75	66.65	8.91	11.14	1.50	.75	70.06	9.36	20.14	2.68
17.0	66.72	8.92	11.34	1.52	29.0	70.14	9.37	20.34	2.71
.25	66.79	8.93	11.52	1.55	.25	70.21	9.38	20.54	2.73

Table Showing the Weight per Cubic Foot and U. S. Gallon—Continued

Degree Brix	Weight of		Solids (Brix) per		Degree Brix	Weight of		Solids (Brix) per	
	1 cu.ft.	1 gal.	1 cu.ft.	1 gal.		1 cu.ft.	1 gal.	1 cu.ft.	1 gal.
	Lbs.	Lbs.	Lbs.	Lbs.		Lbs.	Lbs.	Lbs.	Lbs.
29.50	70.29	9.39	20.74	2.76	42.0	74.18	9.91	31.16	4.16
.75	70.36	9.40	20.93	2.79	.25	74.26	9.92	31.38	4.19
30.0	70.44	9.41	21.13	2.82	.50	74.34	9.93	31.60	4.22
.25	70.52	9.42	21.33	2.85	.75	74.42	9.94	31.82	4.25
.50	70.59	9.43	21.53	2.88	43.0	74.51	9.96	32.04	4.28
.75	70.67	9.44	21.73	2.90	.25	74.59	9.97	32.26	4.31
31.0	70.74	9.45	21.93	2.93	.50	74.67	9.98	32.49	4.34
.25	70.82	9.46	22.13	2.96	.75	74.75	9.99	32.71	4.37
.50	70.89	9.47	22.33	2.99	44.0	74.84	10.00	32.93	4.40
.75	70.97	9.48	22.53	3.02	.25	74.92	10.01	33.15	4.43
32.0	71.04	9.49	22.73	3.04	.50	75.00	10.02	33.38	4.46
.25	71.12	9.50	22.93	3.07	.75	75.08	10.03	33.60	4.49
.50	71.19	9.51	23.14	3.09	45.0	75.17	10.05	33.83	4.52
.75	71.27	9.52	23.34	3.12	.25	75.25	10.06	34.05	4.55
33.0	71.35	9.53	23.55	3.14	.50	75.34	10.07	34.28	4.58
.25	71.43	9.54	23.75	3.17	.75	75.42	10.08	34.50	4.61
.50	71.50	9.55	23.95	3.20	46.0	75.51	10.09	34.73	4.64
.75	71.57	9.56	24.15	3.23	.25	75.59	10.10	34.95	4.67
34.0	71.65	9.58	24.36	3.26	.50	75.67	10.11	35.18	4.70
.25	71.72	9.59	24.56	3.29	.75	75.75	10.12	35.41	4.73
.50	71.80	9.60	24.77	3.32	47.0	75.84	10.13	35.64	4.76
.75	71.88	9.61	24.98	3.35	.25	75.93	10.14	35.87	4.79
35.0	71.96	9.62	25.19	3.37	.50	76.01	10.15	36.10	4.82
.25	72.04	9.63	25.39	3.40	.75	76.09	10.16	36.33	4.85
.50	72.11	9.64	25.60	3.43	48.0	76.18	10.18	36.56	4.89
.75	72.19	9.65	25.81	3.46	.25	76.26	10.19	36.79	4.92
36.0	72.27	9.66	26.02	3.48	.50	76.35	10.20	37.02	4.95
.25	72.35	9.67	26.23	3.51	.75	76.43	10.22	37.25	4.98
.50	72.43	9.68	26.44	3.54	49.0	76.52	10.23	37.49	5.01
.75	72.51	9.69	26.65	3.57	.25	76.60	10.24	37.72	5.04
37.0	72.59	9.70	26.86	3.59	.50	76.69	10.25	37.96	5.07
.25	72.67	9.71	27.07	3.62	.75	76.78	10.26	38.20	5.10
.50	72.74	9.72	27.28	3.65	50.0	76.87	10.27	38.44	5.14
.75	72.82	9.73	27.49	3.68	.25	76.96	10.28	38.68	5.17
38.0	72.90	9.74	27.71	3.70	.50	77.04	10.29	38.91	5.20
.25	72.98	9.75	27.92	3.73	.75	77.12	10.30	39.15	5.23
.50	73.06	9.76	28.13	3.76	51.0	77.21	10.31	39.38	5.26
.75	73.14	9.77	28.34	3.79	.25	77.29	10.32	39.62	5.29
39.0	73.22	9.78	28.56	3.81	.50	77.38	10.34	39.85	5.32
.25	73.30	9.79	28.77	3.84	.75	77.47	10.35	40.09	5.35
.50	73.38	9.80	28.99	3.87	52.0	77.56	10.36	40.33	5.39
.75	73.46	9.81	29.20	3.90	.25	77.64	10.37	40.57	5.42
40.0	73.54	9.83	29.42	3.93	.50	77.73	10.38	40.81	5.45
.25	73.62	9.84	29.63	3.96	.75	77.82	10.39	41.05	5.48
.50	73.70	9.85	29.85	3.99	53.0	77.91	10.41	41.29	5.52
41.75	73.78	9.86	30.07	4.02	.25	77.99	10.42	41.53	5.55
.0	73.86	9.87	30.29	4.04	.50	78.07	10.43	41.77	5.58
.25	73.94	9.89	30.50	4.07	.75	78.18	10.44	42.01	5.61
.50	74.02	9.89	30.72	4.10	54.0	78.26	10.46	42.26	5.65
.75	74.10	9.90	30.94	4.13	.25	78.35	10.47	42.51	5.68

Table Showing the Weight per Cubic Foot and U. S. Gallon—Continued

Degree Brix	Weight of		Solids (Brix) per		Degree Brix	Weight of		Solids (Brix) per	
	1 cu.ft.	1 gal.	1 cu.ft.	1 gal.		1 cu.ft.	1 gal.	1 cu.ft.	1 gal.
	Lbs.	Lbs.	Lbs.	Lbs.		Lbs.	Lbs.	Lbs.	Lbs.
54.50	78.44	10.48	42.75	5.71	67.0	83.06	11.10	55.65	7.43
.75	78.53	10.49	43.00	5.74	.25	83.15	11.11	55.92	7.47
55.0	78.62	10.51	43.24	5.78	.50	83.25	11.12	56.19	7.51
.25	78.71	10.52	43.49	5.81	.75	83.35	11.13	56.46	7.55
.50	78.79	10.53	43.73	5.84	68.0	83.45	11.15	56.73	7.58
.75	78.88	10.54	43.98	5.87	.25	83.54	11.16	57.00	7.61
56.0	78.97	10.55	44.22	5.91	.50	83.64	11.17	57.28	7.65
.25	79.06	10.56	44.47	5.94	.75	83.74	11.18	57.56	7.69
.50	79.15	10.57	44.71	5.97	69.0	83.84	11.20	57.84	7.73
.75	79.24	10.58	44.96	6.00	.25	83.93	11.21	58.12	7.76
57.0	79.33	10.60	45.21	6.04	.50	84.03	11.22	58.40	7.80
.25	79.42	10.61	45.47	6.08	.75	84.12	11.23	58.68	7.84
.50	79.51	10.62	45.72	6.11	70.0	84.23	11.26	58.96	7.88
.75	79.59	10.63	45.98	6.15	.25	84.32	11.27	59.24	7.92
58.0	79.70	10.65	46.23	6.18	.50	84.42	11.28	59.52	7.96
.25	79.78	10.66	46.48	6.21	.75	84.51	11.29	59.80	8.00
.50	79.87	10.67	46.74	6.25	71.0	84.62	11.31	60.08	8.03
.75	79.96	10.68	46.99	6.28	.25	84.72	11.32	60.36	8.07
59.0	80.05	10.70	47.25	6.32	.50	84.82	11.33	60.65	8.11
.25	80.14	10.71	47.50	6.35	.75	84.92	11.34	60.93	8.15
.50	80.24	10.72	47.76	6.38	72.0	85.02	11.36	61.22	8.19
.75	80.34	10.73	48.01	6.41	.25	85.11	11.37	61.50	8.23
60.0	80.43	10.75	48.26	6.45	.50	85.21	11.39	61.78	8.27
.25	80.53	10.76	48.52	6.48	.75	85.31	11.40	62.07	8.31
.50	80.62	10.77	48.78	6.52	73.0	85.41	11.42	62.35	8.34
.75	80.71	10.78	49.04	6.55	.25	85.51	11.43	62.64	8.38
61.0	80.80	10.80	49.29	6.59	.50	85.61	11.44	62.93	8.42
.25	80.89	10.81	49.55	6.62	.75	85.71	11.45	63.22	8.46
.50	80.98	10.82	49.81	6.66	74.0	85.81	11.47	63.51	8.49
.75	81.07	10.83	50.07	6.69	.25	85.91	11.48	63.80	8.53
62.0	81.17	10.85	50.33	6.73	.50	86.01	11.49	64.09	8.57
.25	81.26	10.86	50.59	6.76	.75	86.11	11.50	64.38	8.61
.50	81.35	10.87	50.86	6.80	75.0	86.22	11.52	64.67	8.64
.75	81.44	10.88	51.12	6.83	.25	86.32	11.53	64.96	8.68
63.0	81.54	10.90	51.38	6.87	.50	86.42	11.55	65.26	8.72
.25	81.63	10.91	51.64	6.90	.75	86.52	11.56	65.55	8.76
.50	81.73	10.92	51.90	6.94	76.0	86.63	11.58	65.85	8.80
.75	81.82	10.93	52.16	6.97	.25	86.73	11.59	66.15	8.84
64.0	81.92	10.95	52.43	7.01	.50	86.83	11.60	66.44	8.88
.25	82.01	10.96	52.69	7.04	.75	86.93	11.61	66.73	8.92
.50	82.11	10.97	52.97	7.08	77.0	87.04	11.63	67.02	8.96
.75	82.20	10.98	53.23	7.11	.25	87.14	11.64	67.32	9.00
65.0	82.30	11.00	53.50	7.15	.50	87.24	11.66	67.62	9.04
.25	82.39	11.01	53.77	7.18	.75	87.34	11.67	67.92	9.08
.50	82.49	11.02	54.04	7.22	78.0	87.45	11.69	68.22	9.12
.75	82.58	11.03	54.31	7.25	.25	87.55	11.70	68.52	9.16
66.0	82.68	11.05	54.57	7.29	.50	87.65	11.71	68.82	9.20
.25	82.77	11.06	54.84	7.32	.75	87.75	11.72	69.12	9.24
.50	82.87	11.07	55.11	7.36	79.0	87.86	11.74	69.41	9.27
.75	82.96	11.08	55.38	7.39	.25	87.96	11.75	69.71	9.31